United States Court of Appeals
for the
District of Columbia Circuit

TRANSCRIPT OF RECORD

APPELLANTS' BRIEF.

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA.

NO. 8815.

FREDERICK M. GARFIELD, HERMAN W. DREHER
and WESTERN CARTRIDGE COMPANY,
Appellants,

v.

CONWAY P. COE, Appellee.

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Appeal from the District Court of the United States
for the District of Columbia.

COURT OF APPEALS FOR THE
DISTRICT OF COLUMBIA

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INDEX.

Page

Jurisdictional statement 1

Statement of the case 2

The application and proceedings thereon 4

The trial 5

Statutes and rules involved 10

Statement of points relied upon 12

Summary of argument 13

Argument 14

I. Appellants' written description filed in the Patent Office was a full compliance with the statute and clearly indicated that the novel product might be produced by other methods or procedures than those described in detail as the best known by appellants 14

II. The purpose of the claims is to delineate the essentials of the invention and the claims here involved do so 16

III. The claims here involved are of a style sanctioned by the courts and the Patent Office in cases where the essence of the novelty resides in a new physical form of a well-known material 23

IV. There is a well-recognized distinction between a product invention and the process by which it is made. Where the physical characteristics of the product are defined in a claim the disclosure of the process does not measure the novelty of the product 25

Conclusion 29

Cases Cited.

A. S. Boyle Co. v. Siegel Hardware & Paint Co., 26 F. Supp. 217 21
Anraku v. General Electric Company, 80 F. (2) 958.. 20
B. B. Chemical Co. v. Cataract Chemical Co., 31 F. Supp. 374, 377.................................. 15
Ex parte Gauthier, 48 U. S. Patent Quarterly 330.... 21
Ex parte Holden et al., 49 U. S. Patents Quarterly 216 21
Ex parte Lindstadt, 35 U. S. Patents Quarterly 135..21, 22
Ex parte Welch, 44 U. S. Patent Quarterly 318...... 24
Fruit Growers, Inc., v. Brogdex Co., 283 U. S. 1...... 25
Hide-Ite Leather Co. v. Fiber Products Co., 226 F. 34, 36 (C. C. A. 1).................................. 26
Hill v. Hodge, 12 Appeals, D. C. 528, 1898 C. D. 480.. 17
Holland Furiture Co. v. Perkins Glue Co., 277 U. S. 245, 255 26
Hobbs v. Beach, 180 U. S. 383, 400.................. 23
In re Amtorg Trading Corp., 75 F. (2) 826 (C. C. P. A.) 25
In re Burke, 93 F. (2) 50 (C. C. P. A.).............. 16
In re Clark, 57 App. D. C. 103 (17 F. [2] 862)........ 19
Maurer v. Dickerson, 113 Fed. 870, 51 C. C. A. 494... 26
McBride v. Teeple, 109 F. (2) 789.................. 16
Paper Bag Patent Case, 210 U. S. 405............... 15
Peterson v. Coast Cigarette Vendors, C. C. A. 9, 131 F. (2) 389.................................. 17
Rubber Company v. Goodyear, 9 Wall. 788, 796...... 25
Rubber Company v. Goodyear, 9 Wallace 788........ 19
Rumford Chemical Works v. New York Baking Powder Co., 134 F. 385 (C. C. A. 2)...................... 24
Stevens Pump Co. v. Coe, 67 App. D. C. 191 (91 F. [2] 222) 18

Tilghman v. Proctor, 102 U. S. 707.................. 14
Union Carbide Co. v. American Carbide Co., 181 F. 104 23
Walkup v. Interborough Rpaid Transit Co., 22 F. (2) 266 18
The Wood Paper Patent Case, 23 Wall. 566, 593.....25, 26

Statutes Cited.

R. S. 4886 (35 U. S. C. 31)..........................10, 30
R. S. 4888 (35 U. S. C. 33).....................10, 13, 14
R. S. 4915 (35 U. S. C. 63).........................1, 10
Rules of the Patent Office:
  Rule 34.................................... 11
  Rule 35.................................... 11
  Rule 36.................................... 11
  Rule 37.................................... 11

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APPELLANTS' BRIEF.

JURISDICTIONAL STATEMENT.

This case involves a proceeding under R. S. 4915 (35 U. S. Code 63) against the Commissioner of Patents, to authorize the issuance of a patent on an application filed by Appellants, Frederick M. Garfield and Herman W. Dreher, and assigned to Appellant, Western Cartridge Company. The decision of the Board of Appeals in the Patent Office is found at page 67 of Appellants' Appendix (herein abbreviated App.), the Complaint at page 2, and the Judgment of the Court below at page 95.

STATEMENT OF THE CASE.

The invention here involved relates to an explosive technically named diazodinitrophenol. To shorten this brief, we call it "Dinol."

As cases of this type go, this one is peculiar in that the question of anticipation or the presence or absence of invention over the prior art is not involved. All but two claims of the application were allowed by the Patent Office.

Dinol has been known since 1858. It is now used as an ingredient of explosive compositions utilized in ammunition primers and blasting detonators.

Dinol tends naturally to crystallize in the form of acicular or needle-like crystals, a good illustration (App. 9) of which is Figure 1 of patent 2,103,296 (App. 80). This is the form known since 1858.

The evidence shows that this common form of dinol—needle-like crystals—while a unique and desirable explosive (App. 8), had faults which hindered its use. These faults were:

> 1. It did not flow readily. In the conventional loading operation, the explosive composition is caused to flow through a small orifice. The needle-like crystals of dinol bridge and block the orifice. On a microscopic scale, it is like trying to pour a handful of toothpicks through a funnel (App. 10).
>
> 2. When handled wet it settles very slowly from the liquid vehicle (App. 9).
>
> 3. When handled dry it dusts seriously (App. 9), resulting in:
>
> > (a) Irritation of the respiratory passages of persons exposed to it, causing nose-bleed and sneezing. Extended exposure affects the circulatory system (App. 9-10).
> >
> > (b) Tremendous increase of the hazard of accidental explosions attending its use (App. 9).

The art has long recognized the value of dinol as an explosive but the practical disadvantages attending its use, as enumerated above, so far outweighed its explosive advantages that it was used practically not at all (App. 11, 12, 15). Twelve years ago the Hercules Powder Company undertook the task of obviating the faults of dinol. That effort resulted in the Hancock patent, Re. 19,645 (App. 83), and the Alexander patent, 2,130,296 (App. 79). Dinol thus produced is in the form of "free-flowing relatively smooth pellet-like, discrete particles" (claim 6, Hancock patent). We refer to this type of dinol as the "ball dinol."

Ball dinol was free-flowing (App. 11) and thus obviated the first objection above stated to the natural needle-like crystals. Ball dinol had a higher settling rate (App. 11) than the needle crystals and thus was a partial solution to the second fault above stated. It still dusted (App. 11); hence the comfort and physical well-being of persons working with it continued to be jeapordized, and the hazard of accidental explosions was unmitigated.

But ball dinol did not keep. It was unstable (App. 11). Thus the advantages of the ball dinol were largely nullified by the presence of an objection which did not obtain with the common needle-like crystal. Ball dinol was commercially unusable in ammunition (App. 11, 12).

The problem thus posed for the art was well expressed by witness Franz (App. 12):

> "The problem which faced us was the development of an explosive material which would combine the good properties of dinol as it was then known with physical characteristics which would permit its easy loading."

Garfield and Dreher, the inventors here, in pursuit of that problem, made the surprising discovery that dinol made in the form of tabular* crystals was the complete answer (App. 13, 27).

Tabular dinol was as stable as the common needle form (App. 13, 43). It settled rapidly and compactly (App. 18, 20). It did not dust (App. 22-23). It was as free-flowing as the ball type (App. 13). In short, it had all the advantages of the previously known forms of dinol without the disadvantages of either (App. 29-30).

The Application and Proceedings Thereon.

In the application, two illustrative procedures were disclosed for building the tabular dinol (App. 41-42). Both procedures involves the use of a "crystal growth control ingredient." This is a familiar tool in the chemists' kit. Five suitable crystal growth control agents were named (App. 40), all being dyes.

The resultant product — tabular dinol — was described (App. 39, 42-44) as were its advantageous properties. The first claim of the application, as originally filed, read:

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals" (App. 45).

Claim 1 was amended to its present form (App. 58). Claim 14 was inserted (App. 61). The case was appealed to the Board of Appeals in the Patent Office upon both claims here involved, to wit:

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces."
>
> "14. As a new product, tabular crystals of diazodinitrophenol."

---

*See Exhibit 1 (App. 35) for contrast between the class of crystals called "tabular" and the class of crystals called "acicular" or needle-like.

The Board of Appeals affirmed the rejection upon the premise that Appellants had not disclosed tabular dinol as such; that the only thing disclosed was a tabular crystal of dinol stained with a modicum of dye; and that the dye is as much a part of the crystal as the dinol (App. 67).

The Trial.

In the Court below the proofs were addressed to showing, and unmistakably did show, that the improvements attained were attributable to the tabular form and not to the presence of any dye (App. 16, 21). Comparisons between tabular dinol made in the presence of dye and tabular dinol made in the absence of dye† showed conclusively that the same properties were exhibited (App. 21, 30). Comparisons between tabular dinol made in the presence of dye and nontabular dinol made in the presence of the same amount of the same dye showed conclusively that the latter did not have the advantageous characteristics of the tabular crystal (App. 21, 30).

In these comparisons four samples of dinol were used. One (Ex. 3, App. 70) was tabular dinol made by the process described in the application using a dye (App. 15). Another (Ex. 4, App. 72) was nontabular (cockleburr-like) dinol made with the same ingredients as Ex. 3, using the same amount of the same dye as with Ex. 3 (App. 16). Another (Ex. 5, App. 73) was tabular dinol made without any dye at all (App. 17). The fourth (Ex. 6, App. 74) was the common needle form (App. 17). To demonstrate the free-flowability and settling properties, four bottles, each containing the same amount of dinol in water, were produced (App. 16-17). These were simultaneously shaken to suspend the dinol in water, and then allowed to settle (App.

---

†The procedure whereby tabular dinol is made without dye is not disclosed in the application. It is an inferior method (App. 15).

18). The result of the demonstration is graphically illustrated on the opposite plate. The demonstration is described (App. 18, 20-21) in the record but, for safety, the actual samples were not left with the Court. The demonstration showed that, whether made with or without dye, the tabular dinol samples behaved alike—that they settled faster and more compactly than the nontabular samples whether with or without dye (App. 21).

> "Q. 134. From that demonstration could you say whether it is the presence of the dye or the shape of the crystals which determines the improvement in the properties? A. I consider it is adequate proof that it is not the dye, the presence of the dye, which confers the desirable properties.
>
> Q. 135. Well, it is adequate proof of anything more, in your opinion? A. It is proof that the tabular crystals are usable, whether they contain dye or do not contain dye; and the nontabular materials are not usable in our process, whether they contain dye or do not contain dye."

Furthermore, the cloudiness of the supernatent liquors in the nontabular samples after five minutes settling indicates the presence of fine particles which, if dry, would be dust (App. 21).

To further demonstrate the nondusting properties of tabular dinol (whether made with or without dye), the four samples above mentioned were dry screened and photographed after screening (App. 18-20, 22-23). Exhibits 9, 8, 10 and 7 correspond respectively to Exhibits 3, 4, 5 and 6. In the case of the nontabular crystals (whether with or without dye) extreme fragmentation occurred (App. 19, 22). This indicates that they dust severely (App. 22). On the contrary, the tabular crystals (whether made with dye or without) showed themselves to be nondusting (App. 22-23).

SETTLING DEMONSTRATION

Needle Tabular (No Dye) Non-Tabular (With Dye) Tabular With Dye

AS AGITATED

Suspension Suspension

ONE MINUTE AFTER AGITATION CEASES

Clear Clear

Suspension Precipitate Suspension

Precipitate

FIVE MINUTES AFTER AGITATION CEASES

Reasonably Clear Clear Cloudy

Clear

Settled Solids Settled Solids

These demonstrations did not include the ball type dinol because the material was not available (App. 23). The witness knew from experience that ball dinol did dust (App. 23). He also knew that ball dinol was unstable (App. 12).

These comparisons are summarized (App. 29-30) in that the tabular form of dinol, whether made with a dye or without a dye, is superior to the needle type, in that (1) it is nondusting; (2) it has better settling properties, and (3) it is free-flowing. The tabular dinol is superior to the ball type dinol in that (1) tabular dinol is stable; (2) tabular dinol has improved settling properties over the ball type; (3) tabular dinol is nondusting. These advantages of the tabular dinol over previously known types obtained whether the tabular dinol is made with a dye or without a dye. Nontabular dinol made with a dye has desirable stability properties, but is not free-flowing and is a bad duster.

Despite this evidence that it was the tabular crystal form (and not the dye) which conferred meritorious characteristics upon the product, the District Court dismissed the complaint for the apparent reason epitomized in Finding 6 (App. 94), that "the application * * * does not disclose the **production** of any tabular crystal which does not contain dye * * *."

The passages of the application, as filed, relied upon as adequate to support the two claims here sought, are as follows:

> "This invention relates to explosives, and more particularly to diazodinitrophenol of novel form" (App. 37).
>
> "The present crystalline form is further distinguished by clarity, an excellent degree of purity and stability, and resistance to disintegration into fine particles" (App. 39).
>
> "The diazodinitrophenol produced in accordance with the above example consists of clear green tabular

crystals having, on the average, dimensions of about 0.060 mm. by 0.033 mm. by 0.013 mm. Figure 1 of the accompanying drawing forming part of this specification shows such crystals as they appear by transmitted light under the microscope. The principal crystal faces appear to be substantially rectangular and the crystal edges are frequently beveled so that the cross section perpendicular to the long (App. 42) axis is rectangular, hexagonal, or octagonal in outline, as shown in Figures 2, 3 and 4, respectively. Twinned crystals may be observed occasionally, and the crystal faces and edges may acquire a pitted or etched appearance as a result of extensive washing. The color imparted by the adsorbed dye permits the identification of the crystals.

"These crystals settle rapidly from suspension in liquids to a paste containing 60%-65% solids, flow freely when dry, and, on being poured into a container in this state, have an apparent density of about 0.55 g. per cc.; in contrast, the elongated crystals resulting when a crystal growth control ingredient is not employed during the precipitation, settle slowly from suspension in a liquid to a paste containing 45%-50% solids, tend to form matter lumps when dry, and in this state have an apparent density of about 0.32 g. per cc.

"With regard to stability, these crystals appear to be superior to the prior free-flowing forms of diazodinitrophenol, as illustrated by the comparative results of hot moist storage tests shown in the following table:

	Rounded Sandy Grains	Prior Elongated Crystals	Crystals of This Invention
Original weight	1.000 g.	1.000 g.	1.000 g.
Original instantaneous flash point	210° C.	210° C.	210° C.
After 26 days' storage at 74° C. and 85% relative humidity: Weight	0.835 g.	0.908 g.	0.900 g.
—Instantaneous flash point	260° C. (Weak Explosion)	210° C.	210° C.

"Accordingly, the purity and stability of the novel crystalline diazodinitrophenol render it outstanding for use in detonators and in ammunition priming compositions (App. 43).

"It is to be understood that the procedural details given above are purely illustrative and that a desirable product may be obtained in modifications of the described procedure within the spirit of the invention, which accordingly is not to be limited to such details" (App. 44).

"1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals" (App. 45).

STATUTES AND RULES INVOLVED.

The pertinent portions of the Statutes involved are:

> R. S. 4886 (35 U. S. C. 31). Any person who has invented or discovered any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvements thereof * * * not known or used by others in this country, before his invention or discovery thereof, and not patented or described in any printed publication in this or any foreign country, before his invention or discovery thereof or more than one year prior to his application, and not in public use or on sale in this country for more than one year prior to his application, unless the same is proved to have been abandoned, may, upon payment of the fees required by law, and other due proceeding had, obtain a patent therefor.
>
> R. S. 4888 (35 U. S. C. 33). Before any inventor or discoverer shall receive a patent for his invention or discovery, he shall make application therefor in writing, to the Commissioner of Patents, and shall file in the Patent Office a written description of the same, and of the manner and process of making, constructing, compounding, and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art or science to which it appertains, or with which it is most nearly connected, to make, construct, compound, and use the same; and in case of a machine, he shall explain the principle thereof, and the best mode in which he has contemplated applying that principle, so as to distinguish it from other inventions; and he shall particularly point out and distinctly claim the part, improvement, or combination which he claims as his invention or discovery. The specification and claim shall be signed by the inventor.
>
> R. S. 4915 (35 U. S. C. 63).

The rules of the Patent Office involved are as follows:

> 34. The specification is a written description of the invention or discovery and of the manner and process of making, constructing, compounding, and using the same, and is required to be in such full, clear, concise, and exact terms as to enable any person skilled in the art or science to which the invention or discovery appertains, or with which it is most nearly connected, to make, construct, compound, and use the same.
>
> 35. The specification must set forth the precise invention for which a patent is solicited, and explain the principle thereof, and the best mode in which the applicant has contemplated applying that principle, in such manner as to distinguish it from other inventions.
>
> 36. In case of a mere improvement, the specification must particularly point out the parts to which the improvement relates, and must by explicit language distinguish between what is old and what is claimed as new; and the description and the drawings, as well as the claims, should be confined to the specific improvement and such parts as necessarily co-operate with it.
>
> 37. The specification must conclude with a specific and distinct claim or claims of the part, improvement, or combination which the applicant regards as his invention or discovery.

STATEMENT OF POINTS RELIED UPON.

1. That Findings of Fact Nos. 6, 7, 8, 9, and that portion of Finding of Fact No. 2 which holds that Plaintiffs' application does not suggest the possibility of producing tabular crystals of diazodinitrophenol without dye, were each and all induced by an erroneous view of the law.

2. That the inclusion of original claim 1 in Plaintiffs' application as filed was:

   (a) A suggestion of the possibility of producing tabular crystals of diazodinitrophenol without dye;

   (b) A disclosure of a tabular crystal of diazodinitrophenol which does not necessarily contain dye as an ingredient;

   (c) A proper basis for claims 1 and 14 of said application; and

   (d) A clear indication that the applicants considered the article of manufacture aspect of their invention to derive from and reside in the novel crystal form.

3. That the evidence establishes that the improved results obtained by applicants' product are attributable to the novel crystal form thereof and not to the presence of any dye.

4. That applicant is entitled to product claims, such as claims 1 and 14, which are not limited by the inclusion of dye.

SUMMARY OF ARGUMENT.

1. Appellants' written description filed in the Patent Office was a full compliance with R. S. 4888 (35 U. S. C. 33) and clearly indicated that the novel product (tabular dinol) might be produced by methods or procedures other than the best-known methods or procedures described in detail.

2. Claims appearing in an application as originally filed in the Patent Office constitute a part of the disclosures of such application.

3. The purpose of a claim is not to repeat the disclosure of the invention as set forth in the specification, but to delineate the essentials of the invention and the claims here involved do so.

4. The claims here involved are of a style sanctioned by the Courts and the Patent Office in cases where the essence of the novelty resides in a new physical form of a well-known material.

5. There is a well-recognized distinction between a product invention and the process by which it is made. Where the physical characteristics of the product are defined in a claim the disclosure of the best-known process of making the product does not measure the novelty of the product.

6. Since the application here involved describes the product and the best-known mode of making it, it is immaterial that the application did not also describe an inferior method of making it without dye, particularly since the evidence shows that the desirable properties are attributable to the tabular form of crystal and obtain in spite of, rather than because of, the staining of the surfaces of the crystals with a modicum of dye.

ARGUMENT.

I.

Appellants' Written Description Filed in the Patent Office Was a Full Compliance With the Statute and Clearly Indicated That the Novel Product Might Be Produced by Other Methods or Procedures Than Those Described in Detail as the Best Known by Appellants.

The law (R. S. 4888; Tit. 35, U. S. Code, Section 33) requires that patent applicants shall file in the Patent Office "a written description" of "their invention and of the manner and process of making, constructing, compounding and using it, in such full, clear, concise and exact terms, as to enable any persons skilled in the art or science to which it appertains or to which it is most nearly connected, to make, construct, compound and use the same." Appellants did so. Their written description is indeed more than was necessary to comply with the statute.

The statute goes on to recite that, in the case of a machine, the applicant shall describe the "best mode" in which he contemplates applying the principle. It is, however, the recognized spirit of the statute (compare Patent Office Rule 34) that in describing a product invention the applicant shall be required to describe the **best mode** he knows for making it. Appellants did so (App. 14).

There is no requirement, however, that applicants describe every mode they know for making a new product. Such has been the law from an early date. In Tilghman v. Proctor, 102 U. S. 707, the Supreme Court so held, saying:

> "Perhaps the process is susceptible of being applied in many modes and by the use of many forms of apparatus. The inventor is not bound to describe them all in order to secure to himself the exclusive right to the process, if he is really its inventor or discoverer.

> But he must describe some particular mode, or some apparatus, by which the process can be applied with at least some beneficial result, in order to show that it is capable of being exhibited and performed in actual experience" (l. c. 728, 729).

That doctrine has been repeatedly reaffirmed by the various courts down through the years, as, for example, in the Paper Bag Patent Case, 210 U. S. 405, the Supreme Court said:

> "The invention, of course, must be described and the mode of putting it to practical use, but the claims measure the invention" (l. c. 419).

Appellants' written description (App. 37-48) filed with the Commissioner in this case is an overabundant compliance with the statute, in that they not only describe the "manner and process of making" their tabular crystal of dinol but, in fact, described two illustrative manners and processes, and pointed out that either of these illustrative processes might be further varied by the use therein of any one of five (App. 40) suggested crystallization control agents.

Not only did Appellants' specification describe the **best mode** then known by them for the manufacture of the tabular crystals of dinol, but the process selected by them as best, and hence described, is, in fact, the best known in the art today (App. 14). In but minor respects has it been varied to meet conditions encountered in commercial operations (App. 15).

While in describing their best known way of building tabular dinol Appellants followed "an ordinary practice IN including an example" (B. B. Chemical Co. v. Cataract Chemical Co., 31 F. Supp. 374, 377) of how to make the new product, the application, as filed, plainly indicated that the applicants regarded the product aspect of their invention as extending beyond the example resulting from the illustrative way of building it. No more cogent evidence of this is needed than their original first claim, which read as follows (App. 45):

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals."

Claims in applications as filed are considered as constituting part of the disclosures (In re Burke, 93 F. [2] 50 [C. C. P. A.] and McBride v. Teeple, 109 F. [2] 789).*

This case is, therefore, wholly unlike situations which recently confronted this Court in which, long after the applications were signed and filed by the inventors, the assignees extended the claim to embrace something not indicated in the original application.

The original specification here not only pointed out the virtues of the product and ascribed those virtues to the tabular crystal form, but said "a desirable product" could be made without following the "described procedures," of which "the procedural details given above are purely illustrative" (App. 44).

II.

The Purpose of the Claims Is to Delineate the Essentials of the Invention and the Claims Here Involved Do So.

In addition to the requirement for a written description of the product invention and the process of making it, the statute requires that a patent applicant go a step further and "particularly point out and distinctly claim the part improvement or combination which he claims as his invention or discovery." It is apparent on the face of the statute, therefore, that the office of the claim is to separate the wheat from the chaff in the specification.

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* "It is elementary in patent law that claims contained in an application as originally filed may be considered as a part of the disclosure of the application" (l. c. 796).

In Hill v. Hodge, 12 Appeals, D. C. 528, 1898 C. D. 480, this Court recognized that patent applicants were entitled to make claims which were not restricted to the particular "best mode" set forth in the specification. In that case this Court quoted and approved the statement of the Commissioner, as follows:

> "In respect of this the Commissioner said:
>
> > " 'In making any generic invention the inventor necessarily produces some species of it, and the fact that he has never thought of other specific forms cannot deprive him of his right to a broad claim. The inventor may not regard his invention at the time it is produced as any broader than his specific form, but the question for consideration is not as to his opinion, but is one of fact.'
>
> "There is no doubt of the correctness of this as a general proposition (Roberts v. Ryer, C. D. 1876, 439, 10 O. G. 204, 91 U. S. 150, 157; Stow v. Chicago, C. D. 1882, 131, 21 O. G. 790, 104 U. S. 547, 550; Miller v. Eagle Mfg. Co., C. D. 1894, 147, 66 O. G. 845, 151 U. S. 186, 201)."

The function of the claim is merely to point out the essentials, not to redescribe the invention (Peterson v. Coast Cigarette Vendors, C. C. A. 9, 131 F. [2] 389).

> "The requirements of a patent application are established by 35 U. S. C. A., Sec. 33, which provides that the application shall contain a full description of the invention and 'particularly point out and distinctly claim the part, improvement, or combination' which is claimed as the invention. This only requires that the claims point out the invention, **not that they redescribe it**"* (l. c. 390, 391).

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*Emphasis supplied throughout unless otherwise indicated.

As succinctly said by the Court of Appeals for the Second Circuit (Walkup v. Interborough Rapid Transit Co., 22 F. [2] 266):

> "The means disclosed in a specification may indeed be only as illustration of the invention, not a constituent part of it; but it is exactly the office of the claim to distinguish between illustrative and constituent elements" (l. c. 267).

Were it otherwise, the statutory requirement for a claim would be a vain and useless thing, simply a repetition of what had been said in the specification. Appellants' claims here involved express the essence of their invention, namely, dinol—in the form of tabular crystals. The unessentials are omitted by the claims, which is as it should be according to the statute.

Patent claims seldom, if ever, recite every detail or ingredient included within the description. In making a full and complete disclosure, it is invariably necessary to make reference to environment, to expedients and tools employed in building the invention, but which are mere incidents to its production and not essentials of the thing itself. A good case illustrating this is that of Stevens Pump Co. v. Coe, 67 App. D. C. 191 (91 F. [2] 222). In that case the specification disclosed a rubber diaphragm having a certain flanged construction. This Court, however, held the following claim patentable:

> "1. An expansible hollow pumping member having an internal annular flange at one end and an external annular flange at the other end" (l. c. 222).

The significance here is that the claim did not recite every particular mentioned in the specification or even the material of which the "pumping member" (diaphragm) was made, to wit: rubber.

In Rubber Company v. Goodyear, 9 Wallace 788, one of the patents involved was for the product now known as vulcanized rubber. The only way disclosed by the specification for making vulcanized rubber was to mix the crude rubber with sulphur or white lead or other salts of lead, and coloring matter, then heating the mixture. No way of making the vulcanized rubber without sulphur or equivalent material was shown by the specification, yet the Supreme Court sustained a claim making no reference thereto, as follows (76 U. S. 795):

> "the new manufacture of vulcanized India-rubber (whether with or without other ingredients), chemically altered by the application of heat, substantially as described."

The claims expressed the essence of the invention as a new product omitting all reference to sulphur—the chemist's tool (like the crystal control agent here) by which the product was fabricated in the described mode of making it.

Another case of interest in this connection is that of In re Clark, 57 App. D. C. 103 (17 F. [2] 862), in which this Court was considering an application for a patent claiming a paving or flooring element composed of rubber having certain surface characteristics. The specification in that case did not describe such rubber flooring element separately, but only in connection with an iron base. The Board of Appeals in the Patent Office had held that the applicant was not entitled to claim the flooring element except in combination with the iron base, but this Court approved the ruling of the Commissioner to the contrary, saying:

> "This ruling of the Examiners in Chief was properly reversed by the Commissioner, holding:
>
> > " 'The block is an element of the combination, and I see no reason for objecting to a claim which

> is broad enough to include such rubber block, whether it is connected to an iron base or not. **Obviously, the block has its utility independently of the iron support, and it is immaterial that appellant did not state this fact in his original specification'** " (17 F. [2] 862),

and held the claims patentable.

In Anraku v. General Electric Company, 80 F. (2) 958, the Court of Appeals for the Ninth Circuit was considering a claim for a filament "composed substantially of tungsten" and having certain crystal characteristics. The "tungsten" therein disclosed was said to contain some impurities, including "less than ¾ of 1% of non-metallic material." Obviously by making the reference to tungsten the common variety obtainable in commerce was referred to. It is still permissible to call the material tungsten albeit there is a permissible amount of impuities. Likewise, here it is permissible to call applicant's tabular crystal by the name of "diazodinitrophenol," despite the fact that some dye may be adsorbed on its surface.

While it is true that the tabular crystals resulting from the process—the best mode—described in the Appellants' specification would have some surface stained with the dye employed as the crystallization control agent, the evidence showed beyond the peradventure of a doubt that the dye did not produce the advantages. The desirable properties result from the tabular crystal form, regardless of whether made with the dye (the chemists' tool) or without it (App. 30). The desirable properties are not attained when the dye is used unless the product turns out in tabular form (App. 30).

To claim a new product without limitation to the unessential ingredients described in the "best mode" of the specification is not only a compliance with the statute, but is the only way in which the statutory requirement for the claim can be vitalized. In A. S. Boyle Co. v. Siegel Hardware & Paint Co., 26 F. Supp. 217, the patent in suit disclosed an adhesive composition consisting of five ingredients, yet the claim of that patent recited but three of those ingredients. The claim was sustained over the objection, as here, that the claim included less than the total number of ingredients indicated as best. There, as here, the claim pointed out the essentials but left the unessentials unsaid.

It is commonplace in the Patent Office for such claims to be allowed although they do not include every ingredient referred to in the specification as an incident of describing the best mode of making the product. For example, in Ex parte Holden et al., 49 U. S. Patents Quarterly 216, the Board of Appeals in the Patent Office was considering claims for salt baths used in the treatment of steel. These had been rejected by the Examiner because they did not recite every constituent of the bath, which the specification (in describing the best mode) had included. The Board reversed, saying:

> "It seems to be the Examiner's opinion that the claims for applicants' baths should set forth by name every constituent and the exact proportion of each. We are unable to agree with this conclusion."

Another case is that of Ex parte Gauthier, 48 U. S. Patents Quarterly 330, where the real "novelty" in the claim appeared to reside in the addition of a small percentage of beryllium and zirconium "to magnesium base alloys." The Examiner had thought that the claims should be restricted to the particular magnesium base alloys disclosed in the specification, but the Board reversed. A case of particular significance here is Ex parte Lindstadt, 35 U. S. Patents Quarterly 135, in which the specification disclosed a spreading agent for insecticide sprays composed of four ingredients, including rosin. The claims were silent with-respect to the rosin and the specifications were silent with respect to certain percentage ranges indicated in certain of the claims. But in reversing the Examiner the Board pointed out that certain claims in the application as originally filed indicated the applicant's belief as to the scope of his invention and were entitled to be read as a suggestion that the rosin might be omitted and that the percentages in question were suitable.

> "Appellant points out that in certain of the original claims in this application, substantially the same percentages were given. It is obvious that the specification in the present case is defective in that it does not cover the specific details set forth in all the claims. However, it is clear that **original claims in an application constitute a disclosure** of the invention as well as the specific description and drawings.
>
> "The main objection noted against these three claims as well as claims 33 to 38 is that they are not supported by the specific description. The Examiner contends that the description does not indicate that the spreader could be used without rosin, dextrine or their equivalents and that all of the original claims include rosin as one of the constituents of the spreader and therefore appellant is not entitled to claims which do not contain this element. We do not believe that this contention is sound. It is stated in the description that the rosin acts as a dispersing agent and **does not in any way enter into a chemical combination** with the other ingredients used. In other words, **its action is physical** and not chemical and it is believed that **the omission of this ingredient and its function is purely a matter of choice**" (35 U. S. P. Q. 137).

As in the case just cited, Appellants' use of a dye as a crystallization control agent (a recognized tool in the chemical art) involves a physical and not a chemical action. The evidence conclusively shows that it makes no difference in the characteristics of tabular dinol whether that tool (dye) or some other is used in building the tabular crystal; and it also shows that dinol made with the same tool (dye) does not have the desired properties unless it is tabular. The use or nonuse of the dye "is purely a matter of choice."

The claims here involved* define the essentials of Appellants' product—dinol in tabular form. That tabular dinol is new, and that it is a substantial improvement over the forms of dinol previously known cannot be disputed.

III.

The Claims Here Involved Are of a Style Sanctioned by the Courts and the Patent Office in Cases Where the Essence of the Novelty Resides in a New Physical Form of a Well-Known Material.

When substantial and unpredictable improvements are achieved by making a known substance in a new physical form, it is patentable and is usually claimed in the manner here attempted. For example, in Union Carbide Co. v. American Carbide Co., 181 F. 104, the Court of Appeals for the Second Circuit was considering a patent for "Crystalline Calcium Carbide." Calcium carbide was a previously known material, but the patentee discovered a new physical form which had great advantages of the prior known forms. He claimed it as follows:

> "As a new product, crystalline calcium carbide existing as masses of aggregated crystals substantially as described."**

The claim was sustained.

---

*"1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces."

"14. As a new product, tabular crystals of diazodinitrophenol."

**The words "substantially as described" which were commonly employed in patents granted prior to 1900 do not affect the situation. Such words are to be imputed into all patent claims (Hobbs v. Beach, 180 U. S. 383, 400).

Another comparable case is that of Rumford Chemical Works v. New York Baking Powder Co., 134 F. 385 (C. C. A. 2), where the novelty was in making "baking powder" in granular (as distinguished from powdered) form. The claim sustained read as follows:

> "1. A baking preparation containing phosphoric acid or its compounds in granular condition essentially free from pulverulent phosphatic material, substantially as described."*

A glance at the patents included in this record convinces that, where the invention involves a new physical form of a known material, the style of product claim here involved is conventional. In the Hancock et al. Pat. Re. 19,645 (App. 84) claim 6 reads:

> "Diazodinitrophenol in the form of free-flowing, relatively smooth, pellet-like, discrete particles."

In the Kilburne et al. patent 1,962,080 (App. 78) claim 4 reads:

> "4. As a new product, tabular crystals of anhydrous disodium phosphate."

In the O'Neil patent 1,627,863 (App. 89) claim 4 reads:

> "A solidified drop of ammonium nitrate."

In the Welch patent 2,188,771 (App. 85) claim 3** reads:

> "An electrode wire consisting essentially of nickel, the grains of which are equi-axed."

---

*The words "substantially as described" which were commonly employed in patents granted prior to 1900 do not affect the situation. Such words are to be imputed into all patent claims (Hobbs v. Beach, 180 U. S. 383, 400).

**This claim was allowed by the Board of Appeals in the Patent Office (Ex parte Welch, 44 U. S. Patent Quarterly 318). The Board held:

> "The examiner also thinks that applicant is merely claiming an electrode made of pure nickel which is a natural product. However, this is not correct because applicant treats the nickel alloy in a certain way so as to obtain very small equi-axed grains. This product is not a natural product."

That the claims here involved:

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals, characterized by a pair of parallel substantially rectangular principal faces.
>
> "14. As a new product, tabular crystals of diazodinitrophenol,"

follow the pattern sanctioned in such cases is apparent.

IV.

There Is a Well-Recognized Distinction Between a Product Invention and the Process by Which It Is Made. Where the Physical Characteristics of the Product Are Defined in a Claim the Disclosure of the Process Does Not Measure the Novelty of the Product.

> "The distinction between product and process patents is clear and is quite generally understood. The product patent is upon an invented or discovered article; the process patent is upon a method of making an article. It not infrequently happens that in the same letters patent invention is recognized in both the article and the method of making it" (In re Amtorg Trading Corp., 75 F. [2] 826 [C. C. P. A.]).

Whether the application, here involved, contains a full written description of how to produce a tabular crystal of dinol without using dye is immaterial. Neither the Statutes nor the Patent Office rules require more of a written description than:

> (a) That the new product (manufacture†) be described; and
>
> (b) That the best known manner of making or constructing it be described.

---

†The term "manufacture" as used in R. S. 4886 (35 U. S. Code 31) is synonymous, and used interchangeably, with the more common word "product" (Cf. The Wood Paper Patent Case, 23 Wall. 566, 593; Rubber Company v. Goodyear, 9 Wall. 788, 796; Fruit Growers, Inc., v. Brogdex Co., 283 U. S. 1).

When the District Court ruled that Appellants' application did not:

> "disclose the **production** of such crystals without the use of a dye" (Finding of Fact 2, App. 93);
>
> "disclose the **production** of any tabular crystal which does not contain dye as an ingredient" (Finding of Fact 6, App. 94),

it manifestly confused process ("production") disclosure with product disclosure. The product invention is distinct from the process aspect. The description of one does not necessarily embrace the other (Holland Furniture Co. v. Perkins Glue Co., 277 U. S. 245, 255). When the product (manufacture) is new it is of no importance how or from what the product was made (The Wood Paper Patent Case, 23 Wall. 566, 593). Appellants' product is distinctive in form and properties. The claims define the physical characteristic—tabular—by which it may be recognized and identified. Such product claims are not to be limited to the process of "production" described in the specification.

> "It is a well-recognized rule that a new product, having definite characteristics by which it may be identified, and which distinguish it apart from the process by which it was produced, may be properly described by such characteristics, and when so claimed and described the claims are not limited to the process. Maurer v. Dickerson, 113 Fed. 870, 51 C. C. A. 494" (Hide-Ite Leather Co. v. Fiber Products Co., 226 F. 34, 36—C. C. A. 1).

The lower Court's confusion of the product invention with the process-of-making-it aspect is even more apparent in Finding of Fact 7 (App. 94), for there the Court applies the **product** claims here involved "to the **process** disclosed in such application."

Furthermore, the consideration (Finding 6) of the dye as an "ingredient" of the crystal suggests that the District Court did not appreciate that the dye was merely adsorbed on the surface of the crystals (App. 38, 43) and not even absorbed by them. While the dye is, in truth, an ingredient of reaction mixture (amounting to less than one-tenth of 1 per cent thereof‡) used in the described best mode of building the tabular dinol crystals, the dye is, in no substantial sense, an "ingredient" of the crystals. Withal the proof shows that the desirable properties are conferred by the tabular form in spite of the presence of a modicum of dye.

When Findings 8 and 9 are considered in the light of the other findings it is evident that they, too, were arrived at through confusion of the process with the product aspect of the invention. The product defined by Claims 1 and 14, here involved, was disclosed by original Claim 1 of the application:

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals" (App. 45).

It was disclosed by the drawings (App. 51) read in connection with the following passages of the specification:

> "Figure 1 of the accompanying drawing forming part of this specification shows such crystals as they appear by transmitted light under the microscope. The principal crystal faces appear to be substantially rectangular and the crystal edges are frequently beveled so that the cross section perpendicular to the long axis is rectangular, hexagonal, or octagonal in outline, as shown in Figures 2, 3 and 4, respectively" (App. 42-43).
>
> "These crystals * * * flow freely when dry * * *" (App. 43).

---

‡In Example I of the specification (App. 41) the weight of the reaction mixture exceeds 8,000 grams including 6 grams of dye. In Example II (App. 42) the reaction mixture exceed 8,000 grams and contains 5.2 grams of dye.

Thus the new product, and all the characteristics by which it is defined in Claims 1 and 14, is described. That, plus a description of the best mode of building the new product, is all that is required to constitute a proper basis for the product claims.

While some of the faces of the crystals (resulting from the best-known processes) may be stained with dye so that the material cannot be said to be chemically pure,* no one can deny that such crystals substantially consist of diazodinitrophenol; no one can deny that they are pure enough to serve every purpose which diazodinitrophenol has ever served **and then some:**

> "Q. 75. Have you been able to do anything with this tabular crystal form of dinol that could not have been done with the ball or needle or, for that matter, any other known type of dinol? A. We consider that we have. This dinol has permitted the development of a priming composition which we could turn over, lock, stock and barrel, to unskilled operators in a plant not under our control.
>
> Q. 76. Why couldn't you turn over to such unskilled operators compositions involving the other types of dinol? A. Where the use of the needle form of dinol was involved, we considered that we could use it if we were to use the dinol ourselves; but we would not recommend that it be practiced by anyone who did not have long years of practical experience.
>
> Q. 77. What about the ball type dinol? You said you would not recommend the needle type. A. The ball type dinol was ruled out completely on the question of stability. We could not load that primer in military ammunition" (App. 15).

The original first claim (App. 45) and the last paragraphs of the specification (App. 43-44) amply evidence the inventors' understanding of the product aspect of their invention as extending to tabular dinol, however made, and provide the suggestion, if suggestion be needed, that such tabular dinol might be built in other ways than the described best-known processes.

---

*Few commercial products are chemically pure. No municipal water supply is chemically pure, but they are all pure enough to be accurately described as "water."

The District Court specifically found as a fact that Appellants' application disclosed tabular dinol:

> "2. The Garfield and Dreher application discloses the production of tabular crystals of diazodinitrophenol. * * *" (App. 93).

This is all Appellants are seeking to cover by their claims here in issue. The claims sought are, therefore, consistent with the disclosure.

CONCLUSION.

The application, as filed, clearly indicated that the product aspect of the invention was a tabular crystal of dinol—however made. While the process aspect of the invention was confined to the use of dyes as crystallization control agents—no process claims are in controversy here.

The lower Court, although recognizing that Appellants' application discloses tabular crystals of diazodinitrophenol, apparently considered the patentability of the product to be limited by the disclosure of the applicant's best-known mode of building the product. The law required no more than a disclosure of the product and the best mode of making it. Appellants' compliance was abundant. The presentation of the original first claim was a clear suggestion that the inventors contemplated a product which might be made without dye.

To deny claims, particularly pointing out the essential product novelty, because in the described best-known process of building it the product would be stained with dye, is tantamount to a denial of all protection upon the product. It amounts to restricting a patent on a product to the described best-known process of making it. It is equivalent to striking the word "manufacture" out of R. S. 4886 (U. S. C., Tit. 35, Sec. 31) and abolishing the right to patent new products except insofar as the mode of making them may be protected as an "art."

And finally, with all the unpredictable humanitarian and economic benefits which have been shown to arise from the discovery of tabular dinol, there is no cogent reason why these Appellants should be denied a claim for "a new product, tabular crystals of diazodinitrophenol," when the District Court specifically found that Appellants' application "discloses the production of tabular crystals of diazodinitrophenol" (Finding 2, App. 93).

Wherefore it is submitted that the judgment below should be reversed.

Respectfully submitted,

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APPENDIX TO BRIEF FOR APPELLANTS.

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA.

NO. 8815.

FREDERICK M. GARFIELD, HERMAN W. DREHER
and WESTERN CARTRIDGE COMPANY,
Appellants,

v.

CONWAY P. COE, Appellee.

Appeal from the District Court of the United States
for the District of Columbia.

UNITED STATES
COURT OF APPEALS FOR THE
DISTRICT OF COLUMBIA

FILED SEP 15 1944

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INDEX TO APPELLANTS' APPENDIX.

Page

Complaint 2

Answer 5

Trial proceedings 6

Witness, Arvel O. Franz, direct examination....... 6

Cross-examination 30

Plaintiffs' exhibits:

1. Chart showing crystal types.................. 35
2. Certified copy of Garfield et al. application Serial No. 343,706 and proceedings thereon abridged 37
   - Petition .... ,37
   - Specification 37
   - Claims 45
   - Oath of Frederick M. Garfield............. 49
   - Oath of Herman W. Dreher................ 50
   - Drawings 51
   - Patent Office Action, Oct. 3, 1940........... 53
   - Amendment, filed Mar. 24, 1941............ 55
   - Patent Office Action, Jun. 23, 1941.......... 57
   - Amendment, filed Dec. 15, 1941............ 58
   - Patent Office Action, Jan. 26, 1942.......... 60
   - Amendment, filed Apr. 24, 1942............ 61
   - Examiner's Statement to Board of Appeals Nov. 11, 1942.......................... 64
   - Decision of Board of Appeals Feb. 24, 1943.. 67
3. Photograph—Tabular diazodinitrophenol made with dye 70
4. Photograph — Non-tabular diazodinitrophenol made with dye 71
5. Photograph—Tabular diazodinitrophenol made without dye 72
6. Photograph—Needle diazodinitrophenol 73
7. Photograph—Same as Ex. 6, after dusting test.. 73
8. Photograph—Same as Ex. 4, after dusting dust.. 71
9. Photograph—Same as Ex. 3, after dusting test.. 70

10. Photograph—Same as Ex. 5, after dusting test.. 72
12. U. S. patent No. 2,188,771.................... 85
13. U. S. patent No. 1,627,863.................... 89

Defendant's exhibits:
A. U. S. patent No. 1,962,080.................... 75
B. U. S. patent No. 2,103,926.................... 79
C. U. S. patent No. Re. 19,645.................... 83

Findings of fact 93
Conclusions of law 94
Judgment 95
Notice of appeal 96
Statement of points upon which plaintiffs intend to rely on appeal 97

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Appeal from the District Court of the United States
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APPENDIX TO BRIEF FOR APPELLANTS.

In the District Court of the United States
For the District of Columbia.

Frederick M. Garfield, 6012 McPherson Avenue, St. Louis 12, Missouri, Herman W. Dreher, 6226 Loran Avenue, St. Louis 9, Missouri, and Western Cartridge Company, East Alton, Illinois, Plaintiffs, vs. Conway P. Coe, Commissioner of Patents, Washington, D. C., Defendant.	Civil Action No. 20,979. Patent Application Serial No. 343,706.

COMPLAINT.

To the Honorable, The Judges of the United States District Court, for the District of Columbia.

Plaintiffs for their Complaint allege:

1. That Plaintiff, Frederick M. Garfield, is a citizen of the United States and of the State of Missouri, formerly residing at 5578 Cote Brilliante Avenue and presently residing at 6012 McPherson Avenue, St. Louis, Missouri, and Plaintiff, Herman W. Dreher, is a citizen of the United States and of the State of Missouri, formerly residing at 5021-A Oleatha Avenue and presently residing at 6226 Loran Avenue, St. Louis, Missouri, and Plaintiff, Western Cartridge Company, is a corporation organized and existing under the laws of the State of Delaware and having a place of business at East Alton, Illinois.

2. That Defendant, Conway P. Coe, is a citizen of the United States, is Commissioner of Patents of the United States, maintains his official residence within the District of Columbia, and is sued in his capacity as Commissioner of Patents of the United States.

3. That this is an action arising under the Patent Laws of the United States, to-wit, Section 4915 of the Revised Statutes of the United States (Title 28, Section 63, U. S. Code); that no appeal has been taken to the United States Court of Customs and Patent Appeals, and that there are no adverse or opposing parties.

4. That prior to the third day of July, 1940, Plaintiffs, Frederick M. Garfield and Herman W. Dreher, invented certain new and useful improvements in Manufacture of Diazodinitrophenol for which they believed themselves entitled to Letters Patent of the United States; that on July 3, 1940, Plaintiffs, Frederick M. Garfield and Herman W. Dreher, having complied with the statutes of the United States in such cases made and provided, filed in the United States Patent Office a certain application for Letters Patent identified as Serial No. 343,706, said application containing a written description of said improvements in Manufacture of Diazodinitrophenol in such full, clear, concise, and exact terms as to enable any person skilled in the art or science to which it appertains to make, construct, compound, and use the same, and particularly pointing out and distinctly claiming the subject-matter upon which they believed themselves entitled to Letters Patent.

5. That by an instrument in writing recorded in the United States Transfers of Patents on July 3, 1940, Liber G184, page 250, Plaintiffs, Frederick M. Garfield and Herman W. Dreher, did assign unto Plaintiff, Western Cartridge Company, the entire right, title and interest in and to the invention set forth in the aforesaid application and to all rights for Letters Patents therefor, and that Plaintiff, Western Cartridge Company, is under and by virtue of said instrument in writing the owner of the aforesaid application for Letters Patent.

6. That thereafter such proceedings were had that on January 26, 1942, certain claims of the aforesaid application were finally rejected by the Examiner of the Patent Office in charge thereof; that thereafter Plaintiffs on July 20, 1942, did appeal from the decision of the Examiner to the Board of Appeals in the Patent Office, complying in all respects with the rules and statutes in such cases made and

provided; that thereafter on February 24, 1943, said Board of Appeals did render a decision affirming the Primary Examiner and refusing Letters Patent to Plaintiffs upon that subject matter described in said application, as particularly pointed out and distinctly claimed in the following claims:

> 1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces.
>
> 14. As a new product, tabular crystals of diazodinitrophenol.

to all of which Plaintiffs were justly entitled, and that such action of the Board of Appeals resulted in the denial by Defendant of Letters Patent to which Plaintiffs are entitled, all as will appear from a certified copy of the proceedings in the Patent Office upon said application, in Court to be presented.

Wherefore, Plaintiffs demand judgment against the Defendant, the Commissioner of Patents, ordering and requiring him to grant unto Plaintiffs the Letters Patent prayed for under said Serial No. 343,706, on the claims aforesaid, and such other claims germane to the subject-matter as this Court may deem meet.

By John H. Sutherland,
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Charles E. Riordon,
Of Counsel,
852 National Press Building,
Washington, D. C.

ANSWER TO THE COMPLAINT.

To the Honorable the Justices of the District Court of the United States for the District of Columbia:

1, 2, 3. Defendant admits the allegations of paragraphs 1, 2 and 3.

4. He admits that Frederick M. Garfield and Herman W. Dreher, claiming to have invented improvements in the Manufacture of Diazodinitrophenol, filed an application therefor in the Patent Office, as stated in paragraph 4.

5. He admits the allegations of paragraph 5.

6. He admits the allegations of paragraph 6 as to the procedure in the Patent Office. He admits that he has refused and still refuses to issue to plaintiffs a patent containing claims 1 and 14 of the application of Frederick M. Garfield and Herman W. Dreher, Serial No. 343,706, since said claims are deemed to be unpatentable in view of the following prior patents:

Alexander	2,103,926	Dec. 28, 1937,
Hancock, Reissue	19,645	July 16, 1935,
Kilbourne	1,962,080	June 5, 1934,

profert of copies of which is hereby made, and for the reasons set out in the statement of the examiner in answer to the appeal and in the decision of the Board of Appeals of the Patent Office, copies of which will be presented at the trial.

Further answering he avers that this Court is without authority to consider in this action any claims other than those passed upon by the Board of Appeals, to wit, claims 1 and 14, as set out in paragraph 6 of the complaint.

W. W. Cochran,
Solicitor, U. S. Patent Office,
Attorney for Defendant.

October 2, 1943.

I hereby certify that a copy of this Answer to the Complaint was mailed today, October 2, 1943, to the attorney for plaintiffs, Mr. Charles E. Riordon, National Press Building, Washington, D. C.

W. W. Cochran,
Solicitor.

Washington, D. C.,
Tuesday, March 7, 1944.

This cause came on for hearing before the Honorable Daniel W. O'Donoghue, one of the Justices of said Court, sitting in Civil Division No. 6, at 11:40 o'clock a. m., when the following proceedings were had and evidence adduced:

Appearances:

John H. Sutherland, Esq., Charles E. Riordon, Esq., and Lionel E. Goff, Esq., for plaintiffs.

Edwin L. Reynolds, Esq., for defendant.

PROCEEDINGS.

Opening statements were made on behalf of plaintiffs and defendant.

Thereupon

ARVEL O. FRANZ,

called by the plaintiffs as a witness, was first duly sworn by the Deputy Clerk and was then examined and testified as follows:

Direct Examination.

By Mr. Sutherland:

Q. 1. Mr. Franz, will you state your residence and occupation, please? A. I am a resident of Alton, Illinois. My present occupation is assistant research director of The Western Cartridge Company.

Q. 2. Will you speak a little louder? How long have you held the position of assistant research director of The Western Cartridge Company? A. Approximately two years.

Q. 3. Will you give us your educational and practical background which qualifies you for that position? A. I got my bachelor's degree in chemical engineering from Washington University, in St. Louis; and my master's degree, also in chemical engineering, from Lehigh University, at Bethlehem, Pennsylvania.

Immediately after receiving my master's degree I was hired by the Western Cartridge Company as an operator in the high explosives production line. It was understood at that time that the operator's duty was a training for further research work.

Q. 4. Just what do you mean by saying you were an operator? A. I mean that I actually produced the compounds which were then being produced in production for primary explosives.

Q. 5. You mean you were a factory worker? A. Yes.

Q. 6. Go ahead. A. That job lasted something more than a year and a half, during which time I acted as operator on every high explosive on which we were then engaged.

I was transferred then to the ballistics laboratory, at which time I went through a training of approximately one year——

Mr. Sutherland: Pardon me. Does the Court understand what a ballistics laboratory is?

The Court: Yes.

Q. 7. Go ahead. A. ——as a research worker, the object being to further my training for a general research job. As the end of that time I was placed in charge of the high explosives research laboratory, which job I held until approximately 1941.

My work then took me to the chemical engineering phase of carrying on many of the developments of that research laboratory, and assisting in their completion to large scale operations necessitated by the war effort.

Q. 8. In your position in charge of the high explosives research, did you have working under you Mr. Frederick M. Garfield and Mr. Herman W. Dreher, the applicants in this case involved here, or either of them? A. Yes. Mr. Garfield worked under me, under my direction, from December of 1937 until he left the company. Mr. Dreher worked under my direction from that time until some time in 1941, when he was transferred to a job in charge of production.

Q. 9. At the time of 1937 or 1938, you in the laboratory of the Western Cartridge Company were familiar with "dinol", weren't you, as an explosive? A. Yes.

Q. 10. And what are the characteristics of dinol? A. Dinol is——

Q. 11. (Interposing) And, for the sake of the record, when I say "dinol"' what do you understand by that? A. That is diazodinitrophenol.

Q. 12. Now, is there a contraction for that? A. We use "dinol" as a contraction for that.

Q. 13. All right, sir. What are the characteristics of that? A. Dinol is an explosive which combines the properties of an initiating and a secondary explosive. By that I mean that it has the sensitivity of an initiating explosive; but also has the power of a secondary explosive.

Q. 14. What is an example of an initiating explosive and an example of a secondary explosive? A. Mercury fulminate is a well known example of an initiating explosive and TNT is an example of a secondary explosive.

Q. 15. Did you discover the properties of dinol to which you have just referred? A. No. Those properties are well known.

Q. 16. All right. What other properties did dinol have, as you knew it in your laboratory? A. Dinol was available to us then in two forms. the first form it consisted of long needle-like crystals, approaching fiber characteristics. It was also available——

Q. 17. Do such crystals have any particular name in the crystallography art? A. They are referred to as acicular in habit.

Q. 18. A-c-i-c-u-l-a-r? A. Yes.

Q. 19. I call your attention to a sheet (which I will mark Plaintiffs' Exhibit 1) and ask you whether that sheet, on the left-hand side, illustrates the habit of crystals of what you referred to as the needle-like structure? A. On the left-hand side as you hold it, or as I look at it?

Q. 20. On the right-hand side as you look at it. A. That is a representation of the needle-like type of crystals.

Q. 21. All right, sir. Proceed. You said you had two forms of dinol available to you. A. The second form was an agglomerated crystal mass, composed of granules, which was available to us from the Hercules Powder Company.

Q. 22. When you speak of the agglomerated mass, may I ask whether you refer to the form shown in Fig. 2 of the Alexander patent 2,103,296, as a fair general representation of both types that you spoke of? A. Yes; that is a fair representation.

Mr. Sutherland: That is one of the patents, your Honor, that has been handed up to you by counsel for the Commissioner.

Q. 23. Would you say that Fig. 1 of that patent is a fair general representation of the needle-like type of dinol crystals? A. It is.

Q. 24. Now, having those two types of dinol before you for use, what more was there that could be desired? A. Neither one of those types contained all of the properties which would make it desirable, or even useful, for priming ingredients.

First, the needle crystals, for manufacturing purposes, did not settle rapidly. It required a long time to settle sufficiently to permit decanting. The paste to which they settled contained an undesirably large amount of water.

Q. 25. Now, what is the importance of that in the manufacture of explosives, that is, the matter of settling? A. The matter of settling is of importance in two ways: In the first place, the speed of settling limits the speed of manufacture of the material. That is, the washing operations are carried out by permitting it to settle and pouring off the supernatant liquor; and with the needle-like crystals these washing operations take a very long time. The preliminary settling of the solids, that is, the solids to which the paste settles, is of great importance, because it determines the amount of water which the product takes with it to the next stage in the manufacturing operation; and that is a limiting feature. If too much water is involved at that stage, the explosive must be dried before it can be mixed with the other ingredients of the compositions which are used in priming.

Q. 26. Now, did the needle crystals have any other disadvantageous feature? A. In the dry handling which is carried out in certain loading operations, they dusted seriously. This dust was an awful problem. There were physiological reactions to it. There was also considerable danger involved. The dust would float through the air of the room, to be set off by machinery, and would bring about a growth of dangerous particles of the material in places which were supposed to be free of that material.

Q. 27. Did this dust have any particular physiological reaction on the operators? A. Yes. Dinol dust is a pronounced sneeze-producing agent. The operators did not like it. It would irritate the nose and the exudation from the nose would be brownish in color. Extended exposure would produce nose bleed; and an extended exposure would have serious results on the circulatory system—although that is not clearly defined in the medical world. There have not been carried out any extensive investigations, medical investigations, of the properties of dinol. Those properties are enough to make the operators dislike the handling of dry dinol.

Q. 28. Now, with reference to the manipulation of it, either wet or dry? A. Primer mixtures formulated from dinol in the needle crystals form had a property which the operators distinctly did not like. In explaining that, I will have to explain the nature of the operation. In the priming operation, a mass of wet paste is placed on a steel plate, which contains many small holes. This mass is then rubbed into the holes in the steel plate by means of a wooden spatula. This operation, which individually does not take much exertion, continued eight hours a day becomes a tiring operation. Now, the needle-like dinol produced a mix which the operators described as tough; and the reason for this property was that the needle-like form made an interlacing structure.

Q. 29. Like running a box of toothpicks through a funnel? A. Yes. Attempts to remedy that condition by continued mixing to break down the needle-like structure resulted in too many fines and resulted in a condition which the operators described as rubbery or clay-like.

Q. 30. Now, what about the handling of it dry? You said there were some operations in which it was handled dry? A. In the dry charging operations the substantially pure material is brushed into the holes in a similar charge plate. Your illustration is about the best I have heard: it was like trying to pour toothpicks through a funnel. The material mats, nests, and it just will not flow freely and uniformly into the holes of the charge plate. Uniformity must be emphasized.

Q. 31. If I understand you correctly, the needle type of dinol needed improvement in the respect of being free-flowing and needed improvement in the respect of its settling characteristics and also the matter of dusting? A. Yes.

Q. 32. Now, what about the other type of dinol crystals that you knew of at the time? I believe you called it ball dinol. A. The ball dinol did obviate many of these difficulties.

Q. 33. Which ones did it obviate? A. It settled rapidly. Its final settling rate was not as high as could have been desired. It still dusted. But it introduced a new problem, of stability.

Q. 34. Was it free-flowing? A. It was free-flowing in the dry stage.

Q. 35. How about the stability? How did the stability of the ball dinol compare with that of the needle type dinol? A. Extended tests, both of the pure material and of ammunition loaded with it which we made of the pure material showed that this material just did not have adequate stability to justify its use in commercial ammunition.

Q. 36. Now, you have read the Alexander patent 2,103,-926, have you not? A. Yes.

Mr. Sutherland: A copy of that is in your Honor's file.

Q. 37. And the Hancock and Pritchett reissue patent 19,645? A. Yes.

Q. 38. You have read both of those patents? A. Yes.

Q. 39. Now will you state, just generally, to what they relate? A. They relate, in general, to the preparation of a free-flowing material in the form of agglomerated crystals.

Q. 40. And that is what you have referred to as the ball type dinol? A. Yes.

Q. 41. Now will you go a little bit more into detail about what you mean by agglomerated crystals? A. The ball crystal or agglomerated crystal may be made either by individual crystals, which have been formed as individual crystals, and then cemented, by one process or another, into an aggregate of crystals; or it may be the complexly twinned crystals, which are crystals which occur in the form of an aggregate; and while the parts, of which it is made up, belong to a crystal system, the resultant mass is not clearly definable as a single crystal.

Q. 42. Do you still think that Fig. 2 of the Alexander patent is a pretty fair general representation of the type of thing we are talking about? A. Yes.

Q. 43. I see that in the individual particles represented in that figure there are some cross lines? What do they represent? A. I think that is actually the license of the artist in describing by diagrams what he sees. Those would represent the lines along which the crystals came into contact in this aggregate. They are not a perfect representation of either type of the crystals. They are good general representations.

Q. 44. But your experience with the ball type dinol was that it was less stable than the needle type? A. It was markedly less stable than the needle type. We considered it unusable for commercial purposes.

Q. 45. Was it usable for detonators or as a power explosive charge? A. Its only use was in electric detonators.

Q. 46. But not in ammunition? A. It is not found in commercial use, to my knowledge, in ammunition.

Q. 47. Well, now, Mr. Franz, with that level of knowledge in your laboratory, just what was the problem posed for you and Mr. Garfield and Mr. Dreher, who were working under you? A. The problem which faced us was the development of an explosive material which would combine the good properties of dinol as it was then known with physical characteristics which would permit its easy loading.

Q. 48. Did you have any preconceived notion about what character of particle of dinol you might produce to get those desired properties? A. One would normally expect that if you produced a crystal or a mass in which the dimensions in all directions were substantially equal you would improve the properties that we are speaking of. If you produced that as an equant particle——

Q. 49. (Interposing) What would be an example of such a mass? A. A cube or a sphere. If you produced that in the form of a single crystal, one would expect that there would be freedom from the dusting properties.

Q. 50. Then what did you do toward arriving at a solution of that problem? You personally I mean. Did you turn it over to somebody? A. I personally outlined the requirements to Mr. Garfield and Mr. Dreher and told them to see what they could do, to go to work on it.

Q. 51. And, in outlining the requirements, you included, of course, a stability comparable to that of the needle type dinol? A. Yes.

Q. 52. And free-flowing qualities comparable to those of the then known ball type dinol? A. Yes.

Q. 53. Anything else? A. Dusting properties better than either were required; that is, non-dusting properties.

Q. 54. That is, both the known forms of dinol dusted? A. Yes.

Q. 55. And produced these dangerous properties and physiological effects that you mentioned? A. Yes.

Q. 56. Did you tell them anything about the settling properties that you mentioned? A. It had to be equivalent to the ball dinol, at least.

Q. 57. And Mr. Garfield and Mr. Dreher proceeded to try to find the solution of that problem? A. Yes.

Q. 58. Do you know whether they found it? A. They found a solution which was in all ways satisfactory. It was as much as we had hoped for.

Q. 59. What was the solution? A. The solution which they found involved the preparation of the dinol in the form of tabular crystals.

Q. 60. Have you read the application involved in this case? A. Yes.

Q. 61. Serial number 343,706? A. Yes.

Mr. Sutherland: I will mark copy of that application Plaintiffs' Exhibit 2 and hand it up to the Court.

(Certified copy of file wrapper and contents of application in suit was marked as Plaintiffs' Exhibit 2.)

Q. 62. In that application, you know that certain procedures are set forth, whereby these tabular crystals of dinol might be made? A. Yes.

Q. 63. Will you just briefly describe the procedures that are set forth in the specification for making those tabular crystals? A. Do you want the quantities and reagents involved?

Q. 64. Oh, just in a general way. A. Well, the exact quantities involved are a function of the scale of manufacture. I will give approximately the proportions. The approximate proportion is this: To about 440 cc of water are added 45 grams, dry weight, of sodium picramate, and a quantity of dye, approximately 1.5 cc, of what we now designate as a production dyestuff, is added to this mixture. The exact quantity of the production dyestuffs to be used is determined by preliminary small-scale experiments on that lot of dyes.

Q. 65. Because there are variations from lot to lot of dyes? A. There are variations from lot to lot of dyestuffs.

Then a quantity of hydrochloric acid is added; and the exact quantity depends on the titration of the hydrochloric acid then available. After this, sodium nitrite solution is added at a controlled rate.

The temperature of the reaction can be controlled either by an external agent or through the addition of cracked ice to the reacting mixture. The temperature is controlled between 11 and 15 degrees Centigrade.

At the conclusion of the addition of the sodium nitrite, a test is made with starch-iodide paper, which, if it turns blue, shows that you have enough sodium nitrite.

The mother liquor is decanted after a settling period, and the crystals are washed repeatedly by decantation.

Q. 66. You mentioned the use of a dye; and I believe you have read the specification forming part of the application in this case? A. Yes.

Q. 67. The application is in Plaintiffs' Exhibit 2 and at page 5 of that specification there are five different dyes mentioned. Do you know, of your own knowledge, whether all those dyes will work for the purposes that you have described? A. All those dyes will work, but not in exactly the same proportions nor to the same extent.

Q. 68. But they will all work? A. They will all work.

Q. 69. Now, Mr. Franz, at the time this application was filed, will you state whether or not the procedure for making these tabular crystals, as described in this specification, was the best mode that you folks in your laboratory then knew about for making such tabular crystals? A. Yes; it was the best method.

Q. 70. Well, what about today? Is it the best or second best or worst? A. There have been only very minor changes in the concentrations and reaction times. There has been a change from what we then considered the best dye to what we now consider the best dye, although both of them are now on that list.

Q. 71. But the use of one of this series of dyes mentioned here is still the best mode of producing this material? A. Yes.

Q. 72. But you do know now how to make this tabular crystal dinol without any dye at all? A. Yes. That is second best, however.

Q. 73. Have you been able to do anything with—are you now on plant scale production on this tabular crystal dinol? A. Yes.

Q. 74. How does that compare with the process described? A. Almost exactly except scale.

Q. 75. Have you been able to do anything with this tabular crystal form of dinol that could not have been done with the ball or needle or, for that matter, any other known type of dinol? A. We consider that we have. This dinol has permitted the development of a priming composition which we could turn over, lock, stock and barrel, to unskilled operators in a plant not under our control.

Q. 76. Why couldn't you turn over to such unskilled operators compositions involving the other types of dinol? A. Where the use of the needle form of dinol was involved, we considered that we could use it if we were to use the dinol ourselves; but we would not recommend that it be practiced by anyone who did not have long years of practical experience.

Q. 77. What about the ball type dinol? You said you would not recommend the needle type. A. The ball type dinol was ruled out completely on the question of stability. We could not load that primer in military ammunition.

Q. 78. I hand you a photograph, which I will mark as Plaintiffs' Exhibit 3, and ask you to state, if you know, what that shows. A. This shows a sample of tabular dinol, such as is produced in our current plant practice. It is quite a good photograph.

Q. 79. Was that dinol produced in accordance with the process described in this specification? A. Yes.

Q. 80. The Court has a larger picture than you have. Would you state the approximate magnification of the Court's copy? A. About 225 diameters. I think it is that exactly.

Q. 81. Do you have any information, Mr. Franz, which might enable you, as one skilled in the art, to conclude whether the improvement in properties, which you have described as attendant upon the tabular crystal dinol you made, is attributable to the tabular crystal form or to the presence of the dye? A. Yes. We have carried out experiments in which dinol is produced in the presence of exactly the same amount of dye and the product contains the same amount of dye as the tabular form: that material does not exhibit the desirable properties. We have also carried out experiments in which——

Q. 82. (Interposing) Well now, before you leave that one, I hand you a photograph, which I will mark as Plaintiffs' Exhibit 4, and ask you to state, if you know, what that is. A. This shows a sample of dinol produced in the presence of the same amount of dyes, the same proportion of dye, as the tabular form.

Q. 83. That is shown by Exhibit 3? A. Yes.

Q. 84. Well, just what was the difference in the routine of making the non-tabular form shown in Exhibit 4 from the routine of making the tabular form shown in Exhibit 3? A. The temperatures, concentrations, and reaction times were identical. In this sample——

Q. 85. Do I understand that the concentration, the temperature, and the reaction time were identical? A. Yes.

Q. 86. And how about the constituents of the reaction bath? A. They were all the same.

Q. 87. They were identical too? A. They were identical too. The only difference was that in the case of Exhibit 4 the sodium nitrite was added before the hydrochloric acid.

Q. 88. Then there was a difference in the order of the addition of the reagents? A. Yes.

Q. 89. But either way it gave you dinol. Is that right? A. Yes.

Q. 90. And the same amount of dye was present in making Exhibit 4 as in the case of Exhibit 3? A. Yes.

Q. 91. The same dye? A. Yes.

Q. 92. And the same basic ingredients? A. Yes.

Q. 93. Now, with reference to the comparative properties, do you have the data with reference to that, an exhibit or the like? A. I have an exhibit here which demonstrates the settling properties of those and other forms of dinol. (The witness produced glass tubes.) These tubes contain two grams each of four different types of dinol. It is immersed in water and is perfectly safe to handle in this fashion. Each tube contains the same amount of dinol.

Q. 94. What are the four different types contained in those tubes? A. They contain, as sample number 1, needle crystals in which no dye is present; as sample number 2, tabular crystals in which no dye is present.

Q. 95. You said tabular crystals in which no dye is present? A. Yes.

Q. 96. Have you made tabular crystals without dye? A. Yes.

Q. 97. I hand you a photograph marked for identification as Plaintiffs' Exhibit 5 and ask you to state what that shows. A. This is a photograph of tabular crystals which contain no dye.

Q. 98. How does that sample compare with the one that you have in your bottle? A. This is the one I have described here as sample number 2.

Q. 99. In other words, the photograph was taken of part of the same batch from which came the material in that bottle, sample number 2? A. Yes.

Q. 100. Proceed. A. Sample number 3 is called here cockle burr crystals, because of their appearance.

Q. 101. That corresponds with those shown in the photograph, Exhibit 4? A. That corresponds with Exhibit 4.

Q. 102. Yes. A. Sample number 4 is the tabular crystals containing dye.

Q. 103. And that corresponds with photograph Exhibit 3? A. Yes.

Q. 104. Now I hand you another photograph, which I have marked for identification as Plaintiffs' Exhibit 6, and ask you what the material is that is shown in that photograph and how it compares with the samples in your hand. A. This is a photograph of the same material that is in the sample which I have designated here as sample 1.

Q. 105. That is the needle dinol? A. That is the needle dinol. It is representative, it is quite representative, of the product of batches of the needle dinol.

Q. 106. Now, Mr. Franz, can you make a demonstration that may indicate, visually, to the Court, the properties of one type over the other? A. May I demonstrate?

Q. 107. Yes. You are now shaking the four vials together? A. Yes. Now they are completely in suspension. There is no settled material. Now, after one minute, one can see that—I will turn this around so that the numbering shows—you can see the tabular crystals, in sample number 2, settle almost immediately; and——

Q. 108. (Interposing) How can you tell that they settle almost immediately? A. You can see the clear liquor above and you can see the heavy precipitate of crystals below.

Sample 4, which is tabular crystals without dye, settles next most readily.

This sample 2, tabular crystals with dye, settles most readily; you can see that.

The sample 3, containing dye, but not in the tabular form, settles very slowly.

The sample 1, containing no dye, in the needle form, also settles slowly.

By this time the sample number 2, of tabular crystals containing dye, has settled quite a distance.

This sample number 4 has settled almost completely and these samples, numbers 1 and 3, hardly at all.

The second part of the demonstration takes about five minutes; there is a waiting period.

Q. 109. Will you first explain what the result will be and we will proceed with another subject while we are waiting. A. In approximately five minutes the sample of tabular crystals without dye, number 4, will have settled completely; and the tabular crystals with dye, number 2, will also have settled. The latter will have settled to a higher solids content than the tabular crystals without dye. The cockle burr crystals containing dye, number 3, will have settled somewhat; and the needle crystals, number 1, barely at all.

Q. 110. You spoke also of the dusting property; and I believe you stated that both the forms of dinol known in the art at the time you began this investigation were subject to dusting. A. Yes.

Q. 111. Do you have any way of demonstrating to the Court, without making anybody sneeze, how this dusting property of one material compares with another? A. No. That would require the handling of dry material, which would not be safe in this Court. However, photographs have been taken of such an experiment; and the photographs might demonstrate that fact.

Q. 112. I hand you a photograph, which is marked for identification 7; and I will ask you to take that photograph and compare it and show to the Court how that demonstrates the dusting property of needle crystals. You might do better to go up and point to the particles on the Court's copy, which are larger. A. In the sample shown the dry material has been dried, screened through a copper screen, by hand, for two passages through the screen. That is not strictly comparable to the handling which it has in use.

Q. 113. You mean the screen test is not nearly as severe as the actual use? A. No. In actual use the screening involves a "jelly bag" operation. That is a term used in the industry. It is a gauze bag with a string. The screening is accomplished by a conical gauze bag. A conical gauze bag is used, which contains some rubber balls. The apex of the cone is downward and the screening operation is carried out by pulling the string which is attached to the apex of the cone, which moves the cone up and down.

Q. 114. In the case of both your test and the jelly bag operation, is the bouncing of the material on the screen involved? A. Yes; but the jelly bag operation is more severe.

Q. 115. Will you take the Exhibit 7 and show the Court what characteristic of that exhibit indicates the dusting properties of the needle crystals and compare that with Exhibit 6? A. The property which demonstrates the breaking down of these crystals is the reduction in the average size.

Q. 116. That is, between Exhibit 6 and Exhibit 7? A. Yes.

Q. 117. Do you find that reduction in particle size present in the comparison of those two exhibits? A. Yes. I think the measurements will show that the average particle sizes in the photograph of the screened sample, Exhibit 7, is distinctly lower than the average sizes shown in Exhibit 6, the photograph of the unscreened sample.

Q. 118. The photographs show that, don't they? A. Yes.

Q. 119. And the particles broken off, what became of them? A. They are the small specks of dust, which can barely be seen in my magnification.

Q. 120. Will you please take the Court's copy and draw an ink line around some of the particles of dust which you find in Exhibit 7? Can you write on that stuff? A. Not very well.

Q. 121. Point them out, please, to the Court—the dust particles. A. (The witness indicated on Plaintiffs' Exhibit 7.)

Q. 122. Have we had time now, before we go on, to complete your demonstration with the four vials? A. I believe so.

Q. 123. Now you are holding a flash light behind the four vials? A. I will start with number 1.

Q. 124. Number 1 being the needle crystals. A. Number 1 being the needle crystals, which has settled, with a reasonably clear mother liquor, to comparatively low solids content. That is indicated by the height of the column, which shows a substantial height of settled solid material present.

The second has settled with a very clear supernatant liquor and somewhat higher solids content; that is, the height of the settled solid material is lower.

The third has settled with a cloudy mother liquor, too, and solids content just comparable to number 1.

The fourth has settled with a clear mother liquor, too, and solids content which is the highest of any of the samples; that is, the height of the settled solid material is even lower than sample 2.

Q. 125. Well, now, sample number 3, which has not settled, I believe, to any appreciable extent—— A. (Interposing) Yes; it has settled to some extent.

Q. 126. Well, the mother liquor in sample 3 is still quite cloudy, as compared with number 2 and number 4? A. Yes.

Q. 127. That sample 3, you say, is the sample which contains the same amount of dye as the tabular crystals with dye? A. It has been prepared with the same amount. The analysis shows that it contains the same amount of dye.

Q. 128. How about sample number 1, the needle crystals? A. It is somewhat cloudy, cloudier than some of the others, indicating the presence of quite a quantity of fine material.

Q. 129. I notice that the height of the solids in the first vial is, perhaps, twice as great as that of the settled solids in the second vial. Does that feature have any significance at all? A. Since the same weight of material is present in each vial, that is a measure of the settling of the solids.

Q. 130. Is it desirable to have a high column or a low column, given the same weight? A. It is desirable to have the lowest column, which infers a high solids content.

Q. 131. And from the devices with which you have made your demonstration, the columns of solids in the tabular crystals vials, whether made with or without dye, are not over half as high as the column of solids in the vial of needle form crystals. Is that right? A. I think the column in the vial containing the tabular crystals without dye is slightly over half as high as the column of solids in the other one.

Q. 132. Now, the solids in the vial of non-tabular crystals without dye, sample 3, is too high? A. Yes; it is almost identical in height.

Q. 133. With what? A. With the needle crystals without dye, sample 1.

Q. 134. From that demonstration could you say whether it is the presence of the dye or the shape of the crystals which determines the improvement in the properties? A. I consider it is adequate proof that it is not the dye, the presence of the dye, which confers the desirable properties.

Q. 135. Well, is it adequate proof of anything more, in your opinion? A. It is proof that the tabular crystals are usable, whether they contain dye or do not contain dye; and the non-tabular materials are not usable in our process, whether they contain dye or do not contain dye.

Q. 136. Now to proceed some more with the dusting property: this demonstration you have made with the vials does not have anything to do with that? A. The cloudiness of the supernatant liquors infers the presence of fine particles.

Q. 137. You spoke of making some screen tests of the needle like material. Did you make those screen tests with the purpose of demonstrating the dust forming properties? A. Yes.

Q. 138. I hand you a photograph, which is identified as Plaintiffs' Exhibit 8, and ask you to discuss that with reference to the dust forming proclivity. A. This is a photograph of what is here identified as sample 3 of the dry screening. It shows extreme fragmentation.

Q. 139. That compares to the cockle burr crystals, Exhibit number 4, does it not? A. Yes.

Q. 140. Containing dye, but non-tabular? A. Yes.

Q. 141. All right, sir. What indicates the dusting property of that? A. The reduction in the average particle sizes and the presence of very many fine particles.

Q. 142. Indicates that it is a high duster or low? A. It dusts badly.

Q. 143. Now then, from the comparisons of Exhibits 6 and 7 you said that the needle type crystals dusted badly; and from the comparison of Exhibit 8 and Exhibit 4 you say that the non-tabular crystals containing dye dust severely? A. Yes.

Q. 144. What, with reference to the tabular crystals containing dye—and in that connection I hand you a photograph marked Plaintiffs' Exhibit 9—Plaintiffs' Exhibit 9 shows what? A. This photograph is a photograph of the tabular crystals containing dye, with dry screening.

Q. 145. Well then, that compares with Exhibit 3? A. Yes. The fact that there is no apparent fragmentation of the crystals, nor any reduction in average size, nor introduction of any fine particles by the screening operation, indicates the non-dusting nature of the tabular crystals.

Q. 146. As shown in Exhibits 3 and 9? A. That is right; yes.

Q. 147. Now, with respect to the tabular material made without dye, I hand you a photograph marked for identification as Plaintiffs' Exhibit 10; and I will ask you to make the comparison with that. A. This photograph shows a sample of the tabular dinol without dye after screening. It compares, therefore, with the photograph of the tabular crystals before screening.

Q. 148. That is, with Exhibit 5? A. Yes. It also demonstrates, by the clean, sharp appearance of the crystals, the absence of crystal fragments and the fact that there is no reduction in average size, that this tabular material is substantially non-dusting.

Q. 149. Now, we have not made any dusting comparisons before and after screening with the ball type dinol.

The Court: Suppose that we wait until after recess—unless you want to continue.

How much longer do you think the case will take?

Mr. Sutherland: Probably another half hour examination, your Honor.

The Court: We will sit until you finish.

Mr. Sutherland: I imagine there will be some cross-examination.

Mr. Reynolds: It will not be very long.

The Court: He said it will not be very long.

By Mr. Sutherland:

Q. 150. With reference to the ball type dinol, have you made any such screening test to determine the dusting properties? A. The material was not available for testing at this time. We experimented with it. The operators know the dusting properties.

Q. 151. That is, they were subject to sneezing, nose bleed, and such like? A. Yes.

Q. 152. I hand you a photograph, marked for identification Plaintiffs' Exhibit 11, and I ask you to make such statement as you can with reference to what dusting property is shown by that photograph. You can say, first, what it is. A. This is a photograph of a sample of dinol which was received from the Hercules Powder Company in approximately 1939 or the early part of 1940. It shows ball type dinol.

Q. 153. The photograph was made recently or about that time? A. The negative was made at that time. The print was made recently.

Q. 154. Yes. A. This was bought as representative of the free-flowing dinol that we got in experimental quantities from that company and which, as previously stated, tests showed was an unstable material; and the tests also showed that the material dusted badly. This photograph, I think, by itself, without any before or after screening test, indicates the dusting property. You see in the photograph the large crystals, the sandy grains referred to in that patent; and you also see a great many identifiable crystal fragments, broken pieces of crystal, apparently broken off the aggregate material.

The Court: Counsel might want a recess of ten minutes.

Mr. Sutherland: I would like one, your Honor.

The Court: We will come back and finish by 1:30.

(A recess was had, at the conclusion of which the trial was resumed.)

By Mr. Sutherland:

Q. 155. Mr. Franz, do you understand what is meant by the statement "as a new product tabular crystals of diazodinitrophenol"? A. Yes.

Q. 156. And you have read the application forming a part, or, rather, the specification forming a part of the application involved in this case? A. Yes.

Q. 157. Exhibit 2? A. Yes.

Q. 158. Do you find in that specification a disclosure of such a new product, tabular crystals of diazodinitrophenol? A. Yes.

Q. 159. Do you understand what is meant by the expression "As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces"? A. Yes.

Q. 160. And, having read the specification involved here, do you find in that specification a disclosure of that product? A. Yes; I do.

Q. 161. In the decision of the Board of Appeals (which appears at pages 35 et sequitur of the exhibit), the Board makes this statement, commencing at the bottom of page 35 of the file wrapper, Plaintiffs' Exhibit 2:

> "The invention relates to a crystalline diazodinitrophenol in which the crystals are tabular in form, characterized by a pair of parallel substantially rectangular faces. In this form the crystals have the characteristic of being free flowing and stable."

Now, in addition to being free-flowing and stable, there are, as I understand from your previous testimony, other advantages of having a crystal in tabular form. Will you enumerate those additional advantages? A. The additional advantages include the ease of manufacturing operations, the ease of loading the dinol, and the characteristic of settling of a high solids content from water suspension.

Q. 162. And the matter of non-dusting? A. And a non-dusting property in dry handling.

Q. 163. Now I show you a copy of the specification and call your attention to page 3, at line 8, and I will ask you whether or not that context points out the settling advantage which you have just mentioned. A. Yes.

Q. 164. And at line 21 of the same page does that specification point out the non-dusting advantage which you mentioned? A. Yes.

Q. 165. Now, further in the decision of the Board of Appeals, at page 37 of Exhibit 2, commencing at the bottom of page 2 of the Board of Appeals decision, they say:

> "The advantage of having crystals in tabular form was hardly a patentable concept since this crystalline form has been recognized as imparting free flowing characteristics, as shown by Kilbourne, in connection with anhydrous disodium phosphate."

You are familiar, are you not, with the Kilbourne and Booth patent number 1,962,080, referred to in the answer here? A. That refers to disodium phosphate.

Q. 166. That is correct. A. Yes.

Q. 167. And you know that patent purports to describe a process of making disodium phosphate in tabular form? A. Yes.

Q. 168. As well as another form of aggregated crystals? A. Yes.

Q. 169. And you know that patent, at page 2, line 43, says:

> "The physical properties of both the tabular and the aggregated crystals are such as to render them non-caking and extremely free-flowing?"

A. Yes.

Q. 170. Now, Mr. Franz, the Board of Appeals says that:

> "This tabular crystalline form has been recognized as imparting free flowing characteristics."

Do you agree or disagree with that? A. I disagree. There are free flowing tabular crystals. There are tabular crystals which are not free flowing.

Q. 171. Well, limit yourself to the explosives are, in which you work. Do you know of some examples in the explosives art where tabular crystals have been prepared? A. Yes.

Q. 172. Are they always free flowing? A. No. I can think of two examples where they are definitely not free flowing.

Q. 173. Yes, sir. What are they? A. One is tabular crystals of ammonium nitrate. The second is tabular crystals of tetryl.

Q. 174. You have had experience with both? A. Yes.

Q. 175. Both of those materials are explosives? A. Yes.

Q. 176. As a chemist, would you say that the character of diazodinitrophenol would be more nearly comparable to that of ammonium nitrate and tetryl or more nearly comparable to that of disodium phosphate, as is disclosed in the Kilbourne patent? A. It is quite closely related to tetryl and related to ammonium nitrate, in that they are both explosive compounds; but not related to disodium phosphate.

Q. 177. Well now, is the disodium phosphate an explosive? A. No.

Q. 178. From what you say about ammonium nitrate and tetryl being non-free-flowing, although in tabular form, let me ask whether or not, back in 1938, or whenever it was that this program was undertaken, you, as one skilled in the art, would have forecast that a tabular crystal of diazodinitrophenol would have been free-flowing? A. I would not have been willing to predict that any particular crystalline form of any material would or would not be free-flowing,—in advance.

Q. 179. And that is the kind of thing you just have to find out by trial, is it? A. Yes.

Q. 180. Well, back when this program started, did you have any idea about how to make a tabular crystal dinol? A. No.

Q. 181. Would you tell the Court whether or not it was any surprise to you, when Mr. Garfield and Mr. Dreher produced these tabular crystals of dinol, to find that they were so free flowing? A. Well, as I stated previously, it would be expected that the most free flowing form of matter would be something in the nature of cubes or spheres. It was quite a surprise to us to find that tabular crystals possessed, in such a remarkably successful degree, the properties that we were looking for.

Q. 182. Well, Mr. Franz, have you made any crystals of dinol that approached closer to the spherical or cubic than these tabular crystals? A. We have made, by methods closely related to this, crystals of dinol which would approach cubes in their nature.

Q. 183. And you would expect in advance that such a crystal would be more free flowing as it approached closer to a sphere or cube? A. Yes.

Q. 184. Well now, what was your experience, by way of comparison between the tabular crystals of dinol and the near-cubic crystals? A. It was definitely a toss-up, which was the most desirable. They were both, to all intents and purposes, equivalent.

Q. 185. Was that forecast by you, or was that somewhat of a surprise, to find that condition? A. Well, we would expect a cube to be better in its properties.

Q. 186. And you found that it was not. A. We found that there was no substantial difference.

Q. 187. In the Examiner's statement, which appears at page 38 of the Plaintiffs' Exhibit 2, the Examiner points out, on page 3 of his statement, that—

> "Applicant is not the first to have conceived the desirability of having diazodinitrophenol in the form of free-flowing particles."

Of course that is true, isn't it? A. Yes.

Q. 188. The patentee of the ball type wanted that important result too.

And the Examiner goes on to say:

> "Nor did the art need any teaching that tabular crystals of a chemical product would have this desired property since numerous substances have been prepared in the form of tabular crystals as illustrated, for instance, by Kilbourne."

As one skilled in the art, do you consider the Kilbourne and Booth patent a teaching, as the Examiner says, that tabular crystals of a chemical product would have this desired free flowing property? A. No. I consider the teaching that tabular crystals of some products had the desirable free flowing property.

Q. 189. The "some products" being what? A. Being a product which is free flowing in that form.

Q. 190. No. You said that he teaches that tabular crystals of this product are free flowing What product was that? A. In this case disodium phosphate.

Q. 191. And do you consider the Kilbourne patent a demonstration that anything else in the form of tabular crystals would be free flowing? A. No; I do not consider it so.

Q. 192. On the next page of the Examiner's statement, he says:

> "Kilbourne taught the art as to disodium phosphate, a tabular crystal could be obtained in several ways, in at least one of which the presence of the control ingredient was not necessary."

Now, you say you have read the Kilbourne patent? A. Yes.

Q. 194. Do you find in that disclosure several ways of making tabular crystals? A. Only one example of a way of making disodium phosphate tabular crystals is quoted.

Q. 194. There are two examples set forth in the patent, Example I and Example II, while you say only one of them is for making tabular crystals? A. Yes.

Q. 195. Which? A. Example I.

Q. 196. Is there a control ingredient used in that example? A. In the language of the patent itself a crystal control agent is described.

Q. 197. Just read what the patent says. A. Quoting lines 69 to 72, page 1, in Example I:

> "In order that crystallization may be controlled, we find it desirable to add to the solution undergoing evaporation seed crystals of anhydrous disodium phosphate in known manner."

Q. 198. And the other example has reference to what? A. Quoting from page 1, line 85:

> "In order to produce aggregated crystals, we proceed in the same manner as indicated in Example I, above, except that we add to the evaporating solution, or to the solution before evaporation, a small percentage of tetrasodium pyrophosphate."

Q. 199. Do you find there, in connection with Example II, a crystal control agent? A. Yes; in this case, the tetrasodium pyrophosphate.

Q. 200. Well then, would you agree or disagree with the statement of the Examiner that I read, as to Kilbourne teaching the art that tabular crystals could be obtained in several ways? Do you agree or disagree with that? A. He does not give but one example of making tabular crystals. He does not give several ways in which tabular crystals can be produced. The second example says "to produce aggregated crystals."

Q. 201. Then do you agree or disagree with the statement of the Examiner that Kilbourne taught that, while a crystal could be obtained in several ways, in at least one the presence of the control ingredient was not necessary? Do you agree or disagree with that? A. I have to disagree with that one, too.

Q. 202. Because there is a crystal control ingredient used in both examples? A. Yes.

Q. 203. With reference to your current manufacture of tabular crystals or dinol, can you state, by way of comparison, as to the manufacturing cost of tabular dinol as compared with the needle or ball type dinol? A. The manufacturing cost of tabular dinol is definitely higher than that of the needle type dinol.

Q. 204. It is definitely higher? A. It is definitely higher. We consider, whatever the cost, it is well worth while in the products which are the end products.

Q. 205. For what reason? A. It permits manufacturing processes that either would not be possible at all or would be possible only with great hazard.

Q. 206. Now, to summarize the comparisons which you have made between the tabular and non-tabular forms of dinol: As I understand your testimony, the tabular form, whether made with a dye or without a dye, is superior to the long-known needle type dinol, in that: (1) it is non-dusting; (2) it has better settling properties; and

(3), and perhaps most important, is the free-flowing character of the tabular dinol. Is that right? A. Yes.

Q. 207. Now, in comparison with the prior known ball type dinol, as I understand your testimony, the tabular type dinol has far improved stability over the ball type dinol. Is that right? A. Yes.

Q. 208. Is there any other advantage over the ball type? A. It settles to a somewhat higher solids content, which eliminates drying in the manufacturing process, and it is non-dusting.

Q. 209. It is non-dusting? A. Yes.

Q. 210. Do those advantages over, first, the needle type and, second, the ball type obtain whether or not the tabular crystal dinol is made with or without a dye? A. The tabular crystals, both with and without dye, show those properties.

Q. 211. What about the dinol made with dye in non-tabular forms? A. Such dinol shows desirable stability but does not show as good non-dusting qualities.

Q. 212. What would you say about free flowability? A. The flowability of the dinol with dye, but non-tabular, is very poor.

Mr. Sutherland: I offer in evidence, on behalf of the plaintiffs, the exhibits which have been identified as numbers 1 to 11, inclusive.

(The exhibits heretofore so identified and marked were received in evidence as Plaintiffs' Exhibits 1 to 11, both inclusive.)

Mr. Sutherland: You may inquire.

Cross-Examination.

By Mr. Reynolds:

XQ. 213. Does the Garfield and Dreher application disclose any process of making tabular crystals without the use of a dye? A. It discloses a process of making tabular crystals.

XQ. 214. The question is: Does it disclose any process of making tabular crystals without the use of a dye? A. No; it does not.

XQ. 215. So that, in every process disclosed by that application the crystals would contain dye, would they not? A. They would contain dye. The quantity would be very small.

XQ. 216. Do you consider that the crystals which contain dye are better in any respect than those which do not include it? A. No.

XQ. 217. Didn't your demonstration indicate that they settled somewhat more quickly without the dye in them? A. Whether they have the dye in them or not, the product is still within our plant specifications.

XQ. 218. But the application does not tell anyone how to make crystals without dye, does it? A. No; it does not.

XQ. 219. Now, why is it that some tabular crystals flow freely and others do not? A. The theory as to the cause of free flowing is rather muddled. In one of the examples cited the tabular crystals are probably not free flowing because of caking properties. In the other example cited, of the tetril crystals, we do not know why the crystals are not free flowing, except that we suppose there is some electrical property of the surface involved.

XQ. 220. Does that substance flow freely in other forms than tabular crystals? A. Yes.

XQ. 221. Does it flow freely in needle-like crystals? A. It flows freely in the form of spherical aggregates.

XQ. 222. As between needle-like crystals and tabular crystals of the same substance, you would expect the tabular crystals to flow more freely, would you not? A. We would expect that there might be some difference in the case of ammonium nitrate. Ammonium nitrate also forms needle-like crystals; but it does not flow freely in either form.

XQ. 223. But if there were any difference it would be in favor of the tabular crystals? A. That I cannot say.

XQ. 224. Do you think that needle-like crystals might in some cases flow more freely than tabular crystals? A. No. But I cannot say that tabular crystals necessarily flow more freely than needle-like crystals.

XQ. 225. But, other things being equal, you would expect them to, wouldn't you? A. Probably they would.

Mr. Reynolds: That is all.

Mr. Sutherland: That is all.

(The witness retired from the stand.)

Mr. Sutherland: That is all the plaintiffs' case, your Honor, except that I would like to offer certain exhibits.

I forecast from counsel's opening statement and, also, a little bit from his cross-examination, that he may argue that, since in the patent application we showed only one way of making these tabular crystals, and that involved the use of a dye—although we disclosed five dyes that would do it—that under our disclosure we could not have a claim that covered the tabular crystals without the use of a dye.

I should like to offer, as examples, other patents granted by the Patent Office which show that claims comparable in scope with the ones asked here have been granted. This is kind of in anticipation of the defense; but the way these cases are handled we have to proceed a little out of order.

I should like to offer, as Plaintiffs' Exhibit 12, the patent to Welch, number 2,188,771.

Mr. Reynolds: We object to it, your Honor.

The Court: Those cases are not connected with the patent in this case?

Mr. Sutherland: One of them happens to be an explosive and another one happens to be a case on a nickel alloy; the pertinence being that in each of these several patents there is only one way of making the thing disclosed, yet a claim is allowed by the Patent Office which is not limited to that one way of making it; it extends to the product disclosed.

The Court: This Court should not be called upon to go through the patents allowed by the Patent Office—what claims were allowed, and specifications, and so on.

That would be asking this Court to take the Patent Office as laying down the law as governing the Court in this case.

Mr. Sutherland: That is quite correct; but I think this Court should, however, take those as persuasive.

The Court: Don't you see that the attorney for the Patent Office could cite a dozen or two dozen others that would be different from the ones that you have cited; and that this Court would have to go through two or three dozen patents that are different, because, forsooth, some of those might be analogous?

No. I will sustain the objection.

Mr. Sutherland: Under Rule 43 (c), I offer to prove, if permitted to do so, that the patent to Welch, number 2,188,771, discloses only one way of making an electrode wire, which, however, is claimed as follows:

> "3. An electrode wire consisting essentially of nickel, the grains of which are equi-axed."

And I further offer to prove, if permitted to do so, that the O'Neal patent, number 1,627,863, discloses but one way of making a grain of ammonium nitrate, which, however, is claimed as follows:

> "4. A solidified drop of ammonium nitrate."

Mr. Reynolds: Your Honor, I do not think he should get in proof this way——

Mr. Sutherland (interposing): Well, I believe the rules of Civil Procedure provide for an offer of proof.

The Court: You are just saying what you are offering to prove; and the contention of the defendant is that it does not prove that.

You are offering this Court a series of other cases which you think are analogous to this. But how could the Court tell from this patent to O'Neal? Just reading the claims or citing the patent, I could not tell what was in that application.

Mr. Sutherland: You could certainly tell what is in the patent as issued.

The Court: I know. But this Court is not going to read two dozen or half a dozen or a hundred other patents that the Patent Office has granted; and so the Court just won't—if you say you are offering it to prove that claims should be granted which do not include the use of these dyes, the Court says it does not prove anything.

Mr. Sutherland: I repeat the offer to prove.

The Court: Very well.

Mr. Sutherland: Continuing with reference to the O'Neal patent, claim 4 reads as follows——

Mr. Reynolds (interposing): If the Court please, I think it would be sufficient to show that the patents were proffered. I do not think that you need read the contents of them into the record.

The Court: You can offer those in evidence. You do not need to take the time of the Court to read from them. You have stated the basis of your offer and I sustain the objection.

Mr. Sutherland: Your Honor, I offer in evidence, as the Plaintiffs' Exhibits 12 and 13, the patent to Welch, number 2,188,771, and the patent to O'Neal, number 1,627,863.

The Court: Have you any evidence, Mr. Reynolds?

Mr. Reynolds: I just want to offer in evidence, as Defendant's Exhibit 1, this folder, consisting of copies of:

A. Kilbourne patent 1,962,080.
B. Alexander patent 2,103,926.
C. Hancock reissue patent 19,645.
D. Print of drawings.
E. Examiner's statement.
F. Decision of the Board of Appeals.

(The document so produced and identified was received in evidence and marked as Defendant's Exhibit 1.)

Thereupon counsel for plaintiffs and counsel for defendant addressed the Court in argument.

(Thereupon the Court took this case under advisement and the instant hearing was concluded.)

PLAINTIFFS' EXHIBIT 1.

24

CRYSTALS

CRYSTAL HABIT

TABULAR

ACICULAR

ISOMETRIC

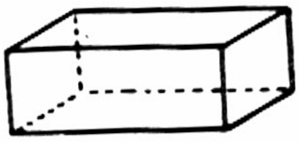


Fig. 52.

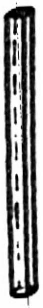


Fig. 55.

TETRAGONAL

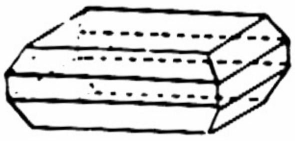


Fig. 57.

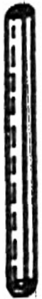


Fig. 60.

HEXAGONAL

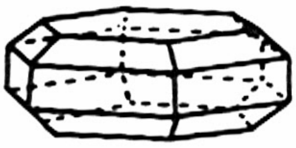


Fig. 62.

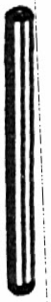


Fig. 65.

ORTHORHOMBIC

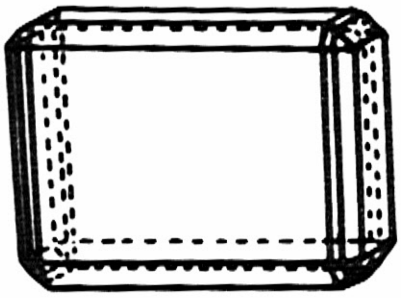


Fig. 67.

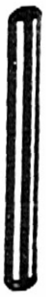


Fig. 70.

MONOCLINIC

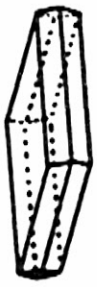


Fig. 72.

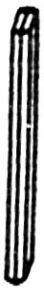


Fig. 75.

TRICLINIC

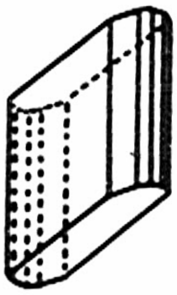


Fig. 77.

Fig. 80.

25

PLAINTIFFS' EXHIBIT 2.

Application Serial No. 343,706, Filed July 3, 1940
(and Proceedings Thereon Abridged).

PETITION.

To the Honorable Commissioner of Patents:

Your petitioners, Frederick M. Garfield and Herman W. Dreher, citizens of the United States and residents of the City of St. Louis, State of Missouri, whose post-office addresses are 5578 Cote Brilliant Avenue, St. Louis, Missouri, and 5021-A Oleatha Avenue, St. Louis, Missouri, respectively, pray that Letters Patent may be granted to them, as joint inventors, for the improvement in

Manufacture of Diazodinitrophenol

set forth in the annexed specification.

And they hereby appoint Joseph Fleischer, c/o Western Cartridge Company, East Alton, Illinois, Register No. 14,374, their attorney, with full power of substitution and revocation, to prosecute this application, to make alterations and amendments therein, to receive the Letters Patent, and to transact all business in the United States Patent Office connected therewith.

Frederick M. Garfield.
Herman W. Dreher.

SPECIFICATION.

To All Whom It May Concern:

Be It Known That We, Frederick M. Garfield and Herman W. Dreher, citizens of the United States, and residing at the City of St. Louis, State of Missouri, have invented the new and useful improvement in

Manufacture of Diazodinitrophenol

of which the following is a specification:

This invention relates to explosives, and more particularly to diazodinitrophenol of novel form and a method for making the same.

Diazodinitrophenol, first prepared and described by Griess in Annalen, Vol. 106, page 123, (1858), has received extensive use in recent years as an ingredient of blasting detonators and ammunition priming compositions. For such use, it is frequently of advantage to utilize a free-flowing form of the explosive. Such a form, consisting of a dark brown, granular powder, was described in Industrial and Engineering Chemistry, Vol. 25, page 664, (1933). Also, the preparation of rounded sandy grains of diazodinitrophenol has been described as resulting from the addition of an acid to a suspension of sodium picramate in a sodium nitrate solution in the presence of an added substance adapted to cause this form of precipitate. However, the free-flowing varieties of diazodinitrophenol heretofore described have consisted of opaque agglomerates of large numbers of small needle-like crystals and/or extremely minute particles.

An object of the present invention is to provide an improved form of diazodinitrophenol consisting of free-flowing crystals.

A further object of the invention is to provide a simple process for the precipitation of diazodinitrophenol wherein crystals are obtained which settle rapidly from suspension in a liquid and which flow freely in the dry state.

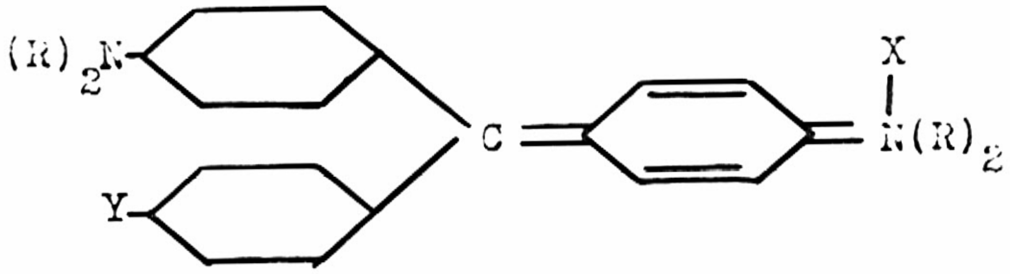
Another object of this invention is to provide a process for the precipitation of diazodinitrophenol wherein desirable crystals of the explosive are produced through the agency of a crystal growth control ingredient.

In accordance with this invention, the precipitation of diazodinitrophenol is carried out by adding a salt of nitrous acid to a solution or suspension of picramic acid, containing an added acid and a small amount of an ingredient having the property of being adsorbed on the crystals as they form in the reaction mixture and thereby of exerting a desirable control on the crystal growth. The resulting diazodinitrophenol precipitate consists of individual crystals having such dimensions as to promote rapid settling from suspension and to impart the property of free flow in the dry state. The action of the crystallization controlling ingredient is such as to prevent growth of the crystals into the elongated columnar or acicular type heretofore known and to promote the formation of crystals having dimensions of length and width of the same order of magnitude.

Accordingly, the process of this invention permits the obtainment of a desirable form of precipitated diazodinitrophenol in the form of individual crystals, as distinguished from the agglomerated free-flowing varieties of the explosive which have heretofore been described. The present crystalline form is further distinguished by clarity, an excellent degree of purity and stability, and resistance to disintegration into fine particles.

Precipitation of the diazodinitrophenol in accordance with this invention may be carried out by preparing an aqueous suspension of picramic acid, as by mixing a picramate such as that of sodium, potassium, ammonium, or magnesium, with an excess of a suitable acid such as hydrochloric, sulfuric, or phosphoric, then adding an ingredient, such as is hereinafter specified, which is adapted to control the growth of crystals as desired, cooling the mixture to a suitable reaction temperature, and then gradually adding a solution of a nitrite, such as sodium or potassium nitrite.

Triphenylmethane dyes having the following structure have been found useful as crystal growth control ingredients:



In this structural diagram, R is an alkyl group, for example methyl, ethyl, propyl or butyl; X is a halogen atom; and Y is a substituted ammonium or amine group. In the following dyes which have been found particularly suitable, R consists of the methyl group, X is chlorine, and Y is as follows:

	Y
Methyl Green	$N(CH_3)_3Cl$.
Ethyl Green	$N(CH_3)_2(C_2H_5)Br$.
Methyl Violet 3 B	$NHCH_3$
Crystal Violet	$N(CH_3)_2$.
Heptamethyl p-rosaniline chloride p-toluenesulfonate	$N(CH_3)_3.OSO_2.C_6H_4CH_3$

Methyl and Ethyl Green, representative of hepta-alkyl p-rosaniline halides are especially advantageous for use in accordance with this invention, for example in the form of their zinc chloride or similar salt addition compounds.

These crystal growth control ingredients are effective for the intended purpose when used in small proportions for example about 0.5% to 2.0% of the weight of picramate employed. The compounds need not be in a pure state, and may therefore be employed as commercially available, generally having a content of inert material. Furthermore, a mixture of two or more of the substances may at times be used to advantage. When a change in the crystal growth control ingredient is contemplated, any modification in the proportion which is made necessary by the change can readily be established by the results of small scale experiments testing the effect of varied amounts.

The dyes listed above are all characterized as displaying a green coloration in acid solution; other triphenylmethane dyes having this property have also been found suitable for use. For example, the application of the reaction product of Malachite Green and methyl p-toluene sulfonate, pentamethyl-diaminotriphenyl chloride p-toluenesulfonate, in the present procedure results in desirable free-flowing crystals of diazodinitrophenol.

It is essential for securing the desired results that the precipitation of the diazodinitrophenol be effected by adding the sodium (or other) nitrite to a mixture of the other reagents (picramic and added acid), the crystal growth control agent being contained either in the latter mixture or partly in the mixture and the remainder in the nitrite solution. Reversal of the precipitation procedure, that is the gradual addition of acid to a mixture of the picramate, nitrite, and dye, results in the precipitation of elongated needle crystals rather than the desirable crystals of this invention.

In order to illustrate the invention, procedural details are given in the following specific examples:

EXAMPLE I.

6.0 g. of Methyl Green, No. 684 in the Colour Index published by the Society of Dyers and Colourists and edited by Dr. F. M. Rowe, 1st Ed. 1924, (biological stain grade containing about 59% of the anhydrous zinc chloride double salt of heptamethyl-p-rosanline chloride) were added to a suspension of 480 g. of pure sodium picramate in 6000 cc. water. The resulting suspension was acidified with a solution of 386. cc. of hydrochloric acid (containing 415.8 g. of HCl per liter) diluted with 480 cc. water, and the mixture was cooled to 11°C. by means of a suitable cooling bath. A solution of 163 g. of sodium nitrite in 720 cc. water was then added uniformly over a period of 25 minutes during continued agitation, the temperature of the reaction mixture being maintained at 11° +1°C. The mixture was then agitated for an additional hour at this same temperature. Agitation was then stopped, the precipitate allowed to settle, and the supernatant mother liquor decanted. The crystalline precipitate was washed three times by suspension with distilled water at a temperature of 5°C., the precipitate being allowed to settle and the wash water removed by decantation in each instance. The product was then stored in the moist state for several hours, when a final cold water wash was applied. The diazodinitrophenol crystals were obtained in a yield of about 400 g. (dry basis), in the form of a paste containing 60%-65% solids.

EXAMPLE II.

To 5500-6500 cc. of water in a suitable container there were added during agitation 480 g. of pure sodium picramate, then 5.2 g. of Ethyl Green, Color Index No. 685, biological stain grade containing about 76% of the anhydrous zinc chloride double salt of ethyl hexamethyl p-rosaniline chlorobromide, and about 410 cc. of hydrochloric acid (containing 415.8 g. of HCl per liter) diluted with 480 cc. of water. The mixture was cooled to a temperature of about 10°C. by means of a suitable cooling bath. During continued agitation, 780 cc. of a solution containing 150 g. of sodium nitrite were added at a uniform rate such that the addition was completed in 25 minutes, the temperature being maintained at 8°-12°C. The reaction mixture was then agitated for another hour at the same temperature. Agitation was then discontinued, the precipitate allowed to settle, and the supernatant mother liquor decanted. The crystalline diazodinitrophenol was then washed and allowed to settle to a paste containing 60%-65% solids, an 85%-90% yield of product being obtained.

The paste of diazodinitrophenol, obtained as described in the above examples, is of desirable consistency for direct use in wet-mixed explosive compositions, and may therefore be stored at constant temperature, in the moist state, until needed. In applications requiring dry explosive, as in the loading of detonators, the diazodinitrophenol produced in accordance with this invention may be dried by any suitable means and yields a product having desirable free-flowing properties.

The diazodinitrophenol produced in accordance with the above examples consists of clear green tabular crystals having, on the average, dimensions of about 0.060 mm. by 0.033 mm. by 0.013 mm. Figure 1 of the accompanying drawing forming part of this specification shows such crystals as they appear by transmitted light under the microscope. The principal crystal faces appear to be substantially rectangular and the crystal edges are frequently bevelled so that the cross section perpendicular to the long axis is rectangular, hexagonal, or octagonal in outline, as shown in Figures 2, 3, and 4 respectively. Twinned crystals may be observed occasionally, and the crystal faces and edges may acquire a pitted or etched appearance as a result of extensive washing. The color imparted by the adsorbed dye permits the identification of the crystals.

These crystals settle rapidly from suspension in liquids to a paste containing 60%-65% solids, flow freely when dry, and, on being poured into a container in this state, have an apparent density of about 0.55 g. per cc.; in contrast, the elongated crystals resulting when a crystal growth control ingredient is not employed during the precipitation, settle slowly from suspension in a liquid to a paste containing 45%-50% solids, tend to form matted lumps when dry, and in this state have an apparent density of about 0.32 g. per cc.

With regard to stability, these crystals appear to be superior to the prior free-flowing forms of diazodinitrophenol, as illustrated by the comparative results of hot moist storage tests shown in the following table:

	Rounded Sandy Grains	Prior Elongated Crystals	Crystals of This Invention
Original weight	1.000 g.	1.000 g.	1.000 g.
Original instantaeous flash point	210°C.	210°C.	210°C.
After 26 days' storage at 74°C. and 85% relative humidity; Weight	0.835 g.	0.908 g.	0.900 g.
—Instantaneous flash point	260°C. (Weak Explosion)	210°C.	210°C.

Accordingly, the purity and stability of the novel crystalline diazodinitrophenol render it outstanding for use in detonators and in ammunition priming compositions.

It is to be understood that the procedural details given above are purely illustrative and that a desirable product may be obtained in modifications of the described procedure within the spirit of the invention, which accordingly is not to be limited to such details.

Having now described the invention, what is claimed as new and is desired to be secured by Letters Patent, is:

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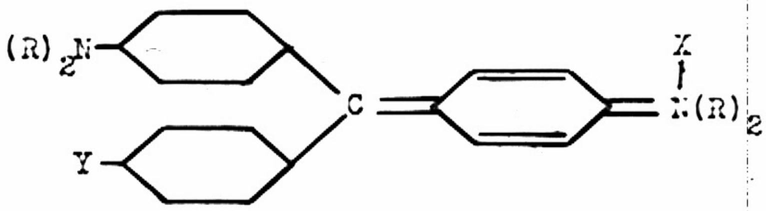
1. As a new product, free-flowing diazodinitrophenol in the form of [illegible] characterized by a [illegible] substantially rectangular principal faces

2. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals containing an adsorbed triphenylmethane dye.

3. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals containing an adsorbed dye having the following structure:



where R is an alkyl group, X is a halogen, and Y is a member of the group consisting of substituted ammonium and amine groups.

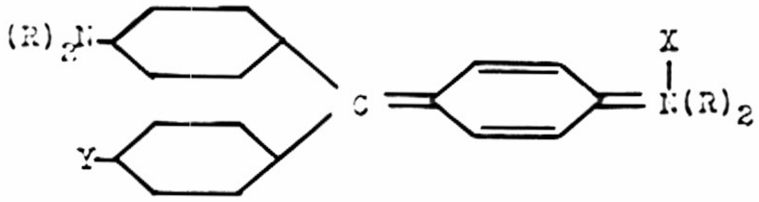
4. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals containing a hepta-alkyl p-rosaniline halide.

5. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals containing ethyl hexamethyl p-rosaniline ~~chlorobormide~~.

6. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals containing heptamethyl p-rosaniline chloride.

7. In the manufacture of diazodinitrophenol, the process comprising gradually adding a nitrite to a mixture of picramic acid and an added acid in the presence of a triphenylmethane dye.

8. In the manufacture of diazodinitrophenol, the process comprising gradually adding a nitrite to a [illegible] picramic acid and an added acid in the presence of a triphenylmethane dye having the following structure:



where R is an alkyl group, X is a halogen, and Y is a member of the group consisting of substituted ammonium and amine groups.

9. In the manufacture of diazodinitrophenol, the process comprising gradually adding a nitrite to a mixture of picramic acid and an added acid, in the presence of a hepta-alkyl p-rosaniline halide.

10. In the manufacture of diazodinitrophenol, the process comprising gradually adding a nitrite to a mixture of picramic acid and an added acid, in the presence of ethyl hexamethyl p-rosaniline chlorobromide.

11. In the manufacture of diazodinitrophenol, the process comprising gradually adding a nitrite to a mixture of picramic acid and an added acid, in the presence of heptamethyl p-rosaniline chloride.

12. A product and ~~process substanti[illegible] as described.~~

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In testimony whereof, we affix our signatures.

Frederick M. Garfield,
Herman W. Dreher.

OATH.

State of Missouri, }
City of St. Louis. } ss.

Frederick M. Garfield, one of the above-named petitioners, being duly sworn, deposes and says that he is a citizen of the United States and resident of the City of St. Louis, State of Missouri; that he verily believes himself and Herman W. Dreher, the other petitioner named herein, to be the original, first and joint inventors of the improvement in

Manufacture of Diazodinitrophenol

described and claimed in the annexed specification: that he does not know and does not believe that the same was ever known or used before their invention or discovery thereof, or patented or described in any printed publication in any country before their invention or discovery thereof, or more than two years prior to this application, or in public use or on sale in the United States for more than two years prior to this application: that said invention has not been patented in any country foreign to the United States on an application filed by them or their legal representatives or assigns more than twelve months prior to this application; and that no application for patent on said improvement has been filed by them or their legal representatives or assigns in any country foreign to the United States: that they have never abandoned the said invention, and that they duly executed the annexed Petition and Specification.

Frederick M. Garfield.

Sworn to and subscribed before me this 27th day of June, 1940.

My commission expires Oct. 8, 1941.

Fannie Forhing,
(Seal) Notary Public.

OATH.

State of Illinois, } ss.
County of Madison. }

Herman W. Dreher, one of the above-named petitioners, being duly sworn, deposes and says that he is a citizen of the United States and resident of the City of St. Louis, State of Missouri; that he verily believes himself and Frederick M. Garfield, the other petitioner named herein, to be the original, first and joint inventors of the improvement in

Manufacture of Diazodinitrophenol

described and claimed in the annexed specification; that he does not know and does not believe that the same was ever known or used before their invention or discovery thereof, or patented or described in any printed publication in any country before their invention or discovery thereof, or more than two years prior to this application, or in public use or on sale in the United States for more than two years prior to this application; that said invention has not been patented in any country foreign to the United States on an application filed by them or their legal representatives or assigns more than twelve months prior to this application; and that no application for patent on said improvement has been filed by them or their legal representatives or assigns in any country foreign to the United States; that they have never abandoned the said invention, and that they duly executed the annexed Petition and Specification.

Herman W. Dreher.

Sworn to and subscribed before me this 29th day of June, 1940.

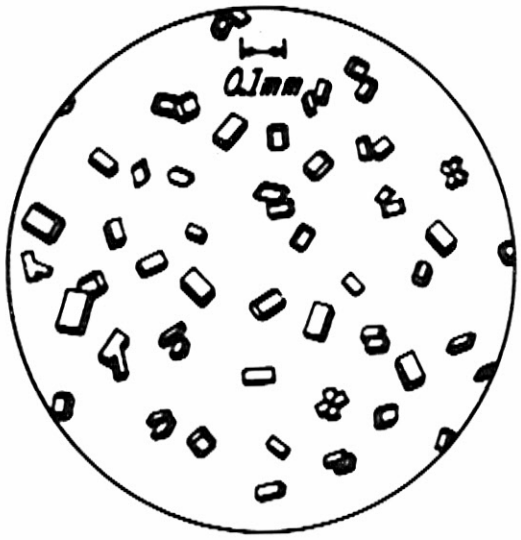
My commission expires July 15, 1942.

Rolla J. Mottaz,
(Seal) Notary Public.

DIV. 43 343706

Print of drawing as originally filed.

Fig. 1



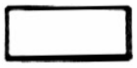


Fig. 2

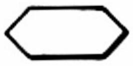


Fig. 3

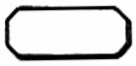


Fig. 4

INVENTORS
Frederick M. Garfield and Herman W. Dreher
BY Jos. Fleischer
ATTORNEY

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(Mailed Oct. 3, 1940.)

Div. 43 Room 4886B Paper No. 3

Department of Commerce
United States Patent Office
Washington

Please find below a communication from the Examiner in charge of this application.

Conway P. Coe,
Commissioner of Patents.

Applicant: F. M. Garfield et al
Ser. No. 343,706
Filed July 3, 1940
For Manufacture of Diazodinitrophenol

Joseph Fleischer
Western Cartridge Co.
East Alton, Ill.

References made of record:

P. Gaubert, Comptes rendus, Vol. 202, pp. 1192-4 (1936) (Pat. Off. Library).

Alexander	2,103,926	Dec. 28, 1937	260-141
Hancock	Re. 19,645	July 16, 1935	260-141

The references are cited to show the state of the art.

Claim 12 is rejected as obviously informal and not in accordance with the requirements of R. S. 4888.

Claim 1 is rejected as incomplete and as failing to define the invention, since it does not point out how the diazodinitrophenol is made free flowing. Claim 1 is further rejected as unpatentable over Hancock or Alexander, which disclose free-flowing diazodinitrophenol, for the reason that the product defined by claim 1 is considered to differ over that of the references only in degree.

Claims 2 thru 11 appear to be allowable as at present advised.

P. J. Federico,
Examiner.

hew;f

MAIL DIVISION
MAR 24 1941
U.S. PATENT OFFICE

U. S. Patent Office
MAR 25 1941
DIVISION 43

IN THE UNITED STATES PATENT OFFICE

FREDERICK M. GARFIELD &
HERMAN W. DREHER — DIV. 43

MANUFACTURE OF DIAZODINITROPHENOL — ROOM 4886B

FILED July 3, 1940 — SER. NO. 343,706

East Alton, Illinois
March 22, 1941

Hon. Commissioner of Patents
Washington, D. C.

Sir:

The Official Letter dated Oct. 3, 1940, was duly received, and the above-entitled application is amended, in response thereto, as follows:

Claim 5, line 3, delete "chlorobormide", and insert therefor --chlorobromide--.

Claim 12, cancel.

Please insert the following claim:

13. As a new product, free-flowing diazodinitrophenol
characterized by a pair of parallel substantially rectangular principal faces and
in the form of tabular crystals containing an adsorbed crystallization-control ingredient.

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par C

R E M A R K S

Reconsideration of this application and allowance of all the claims are respectfully requested since it is believed that the latter define subject matter which is patentable over the prior art.

Claim 1 has been rejected as incomplete and as unpatentable over the product described by Hancock or Alexander. With regard to the first ground of rejection, it is respectfully pointed out that the free-flowing character of applicants' novel

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form of diazodinitrophenol results directly from the fact that the crystals are tabular. As will be seen by reference to Winchell's "Microscopic Characters of Artificial Minerals" (John Wiley & Sons, 1931), page 26, **tabular** applies to crystals in which one pair of opposite faces is decidedly larger than the other faces. Accordingly, it is respectfully submitted that the claim is complete and definitely specifies the invention, since it points out that the desired result (free-flowing character) is secured by providing the diazodinitrophenol in the form of tabular crystals.

With respect to the remaining ground of rejection, it will be seen from study of the descriptions that the present applicants have successfully accomplished the production of free-flowing diazodinitrophenol in the form of clear individual crystals, whereas the product of Hancock or Alexander consists of irregular opaque agglomerates. By producing **tabular crystals** of diazodinitrophenol, applicants have been able to combine maximum stability with desirable free-flowing qualities. Applicants' product is respectfully urged as differing in kind rather than in degree from that of Hancock or Alexander, this difference being fully as striking, for example, as that between the products shown in Alexander's Figures 1 and 2.

Allowance of claim 1 is therefore respectfully urged since it is believed to define accurately a product which is neither anticipated nor suggested by prior art and the advantageous properties of which are described in detail in the specification.

Claim 13, inserted by this amendment, is similar to claim 1, but specifies in addition the presence in the crystals of adsorbed crystallization-control ingredient. In view of the novel character and advantages of applicants' product as defined, allowance of this claim is likewise respectfully solicited.

The Gaubert article, which deals with the modification of uric acid crystals, was cited to show the state of the art, and is not pertinent to the claimed subject matter of applicants' claims.

The allowability of claims 2 through 11 has been noted, and, in view of the foregoing remarks, reconsideration of the application and allowance of all the claims are respectfully requested.

Respectfully submitted,

Frederick M. Garfield and
Herman W. Dreher,
By Jos. Fleischer,
Attorney.

(Mailed Jun. 23, 1941.)

Div. 43 Room 4886B Paper No. 5

Department of Commerce
United States Patent Office
Washington

Please find below a communication from the Examiner in charge of this application.

Conway P. Coe,
Commissioner of Patents.

Applicant: F. M. Garfield et al
Ser. No. 343,706
Filed July 3, 1940
For Manufacture of diazodinitrophenol

Joseph Fleischer
Western Cartridge Co.
East Alton, Ill.

Responsive to amendment filed Mar. 24, 1941.

The rejection of claim 1 as incomplete and as failing to define the invention is adhered to as sound and is repeated. New claim 13 is rejected for the same reasons, as set forth in detail in the last Office action. Claim 1 fails completely to point out how the free-flowing property is imparted. Claim 13 merely adds a functional definition of the ingredient which produces this result.

Claim 13 is further rejected as too broad in claiming a "crystallization-control ingredient," the disclosure being limited to triphenylmethane dyes.

Claims 2 thru 11 stand allowable.

P. J. Federico,
Examiner.

hew:f

In the United States Patent Office

Frederick M. Garfield & Div. 43
Herman W. Dreher

Manufacture of Diazodinitrophenol Room 4886B

Filed July 3, 1940 Ser. No. 343,706

East Alton, Illinois
December 11, 1941

B—Filed Dec. 15, 1941
Division 43

Hon. Commissioner of Patents
Washington, D. C.

Sir:

Responsive to the Official Letter dated June 23, 1941, the above-entitled application is hereby amended, as follows:

Claim 1, line 2, after "crystals", insert **—characterized by a pair of parallel substantially rectangular principal faces—**.

Claim 13, line 2, after "crystals", insert —, **characterized by a pair of parallel substantially rectangular principal faces and—**.

REMARKS.

Reconsideration is respectfully requested of the rejection of claims 1 and 13, which is believed to be obviated by the present amendment. The remaining claims in the case, 2 through 11, have been stated to be allowable.

With regard to the rejection of claims 1 and 13 as incomplete and as failing to define the invention, applicants respectfully submit that these claims accurately describe a form of diazodinitrophenol which is novel and advantageous. Tabular crystals inherently possess the property of free flow because of their shape, since, having dimensions of length and width of the same order of magnitude, (Specification, page 3, lines 7-14), they have no tendency to bridge or become matted together.

Disclosure of applicants' product as "tabular crystals characterized by a pair of parallel substantially rectangular principal faces" is contained on page 7 of the Specification, lines 6-15 and in the drawing. Since the term "tabular" accurately defines one of the several known classes of crystal shapes (as shown by the definitions on page 26 of Winchell's "Microscopic Characters of Artificial Minerals," 1931), distinguishing clearly for example from crystals which may be equant, columnar, or lamellar, and since crystals of diazodinitrophenol having the form specified in these claims are novel, it is believed that claims 1 and 13 clearly point out important aspects of applicants' contribution to the art and should therefore be allowed.

Since crystals of diazodinitrophenol of the specified form constitute a new product, the addition of a further descriptive limitation, corresponding to the disclosure, should not render claim 13 unpatentable. Even if this additional description were functional, it should not affect the patentability, since the expression is not depended upon to differentiate the claimed product from the prior art, that clearly having been taken care of in the remainder of the claim.

With regard to the rejection of claim 13 as being too broad in specifying a "crystallization-control ingredient", it is pointed out that the disclosure is co-extensive; see, for example, page 2, lines 1-14.

It is therefore respectfully requested that the claims be allowed and the case passed to issue.

Respectfully submitted,

Frederick M. Garfield &
Herman W. Dreher,
By Jos. Fleischer,
Attorney.

(Mailed Jan. 26, 1942.)

Div. 43 Room 4886B Paper No. 7

Department of Commerce
United States Patent Office
Washington

Please find below a communication from the
Examiner in charge of this application.

Conway P. Coe,
Commissioner of Patents.

Applicant: F. M. Garfield et al
Ser. No. 343,706
Filed July 3, 1940
For Manufacture of diazodinitrophenol

Joseph Fleischer
Western Cartridge Co.
East Alton, Ill.

Responsive to amendment filed Dec. 13, 1941.

Applicants' arguments have been carefully considered, but nevertheless, the rejection of claims 1 and 13 as functional and as incomplete in the definition of the crystallization control ingredient, is adhered to as sound and is repeated. Since an issue as to the patentability of these claims appears to have been reached, their rejection is accordingly made Final.

Claims 2 thru 11 are allowable.

The final rejection herewith closes the prosecution of the case before the Primary Examiner. Any amendment or request for reconsideration which on inspection is found to require reexamination of the case will not be entered, but, if promptly filed, may be given consideration a reasonable time prior to the expiration of the statutory period. (See ex parte Peterson, 1928 C. D. 31).

P. J. Federico,
Examiner.

hew;f

In the United States Patent Office

Frederick M. Garfield & Herman W. Dreher	Div. 43
Manufacture of Diazodinitrophenol	Room 4886B
Filed July 3, 1940	Ser. No. 343,706

East Alton, Illinois
April 22, 1942

Filed April 24, 1942

Hon. Commissioner of Patents
Washington, D. C.

Sir:

In response to the Official Letter dated Jan. 26, 1942, the above-entitled application is hereby amended, as follows:

Cancel claim 13, and substitute therefor:

14. As a new product, tabular crystals of diazodinitrophenol.

Remarks.

Reconsideration of the final rejection of claims in this application other than allowable claims 2-11 inclusive is respectfully requested, since it is believed that the patentability will be clearly apparent in view of the following.

Claim 1, as follows,

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces."

stands rejected "as functional and as incomplete in the definition of the crystallization control ingredient."

Applicants' showing in Paper No. 6, dated Dec. 13, 1941, to the effect that the above claim accurately defines a novel form of diazodinitrophenol, which has the advantageous property of free flow due to the form specified in the claim, has not been refuted.

Support for applicants' contention may be found in U. S. Patent 1,962,080, issued June 5, 1934 to Kilbourne and Booth, in which the product specified in claim 4, for example,

> "4. As a new product, tabular crystals of anhydrous disodium phosphate."

is distinguished patentably from the prior art product in the free-flowing and non-caking property (Page 2, lines 43-45).

It is also respectfully submitted that the term "tabular crystals" is even more accurate and definite than the expression "relatively smooth, pellet-like, discrete particles" appearing in claim 6 of Reissue Patent 19,645, cited against this application.

Applicants therefore respectfully urge that claim 1 is neither functional nor incomplete, but on the contrary, clearly defines novel subject matter. Claim 14, drawn to the scope of original claim 1, is likewise submitted as directed to patentable subject matter.

As stated by the Court in Binney & Smith Co. v. United Carbon Co., 52 USPQ 205,

> "We entertain no doubt as to the patentability of the product here involved. What we have is not an obvious change of form which anyone could make, once the desirability of such change was recognized, as was the breaking up of the flakes of glue in Glue Co. v. Upton, 97 U. S. 3, but the manufacture of an entirely new form of a product, having new characteristics and advantages, and solving problems in connection with the use of the product which had long baffled those most highly skilled in the art." (l. c. 208),

and, in answer to the contention that certain claims were functional,

> "The patentees had made a substantial contribution to an important art by solving a problem which had baffled other technological experts who had addressed

> themselves to it. They described their product in terms by which any one could identify it; and they set forth a process by which it could be manufactured." (l. c. 209).

In view of the above, it is believed that claims 1 and 14 are in condition for allowance and such action is respectfully requested.

Respectfully submitted,

Frederick M. Garfield &
Herman W. Dreher,
By Jos. Fleischer,
Attorney.

(Mailed Nov. 11, 1942.)

Paper No. 12.

United States Patent Office.
Richmond, Virginia.

In re application of Frederick M. Garfield and Herman W. Dreher. Serial No. 343,706. Filed July 3, 1940. For: Manufacture of Diazodinitrophenol.	Before the Board on Appeal.

EXAMINER'S STATEMENT.

Applicant appeals from the final rejection of claims 1 and 14 (substituted for claim 13 by the amendments after final rejection). Claims 2 to 11 have been allowed. The appealed claims read as follows:

> 1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces.
>
> 14. As a new product, tabular crystals of diazodinitrophenol.

The following references are of record as showing the state of the art:

Alexander	2,103,926	Dec. 28, 1937
Hancock	Re. 19,645	July 16, 1935
Kilbourne	1,962,080	June 5, 1934.

The subject matter at issue relates to the preparation of diazodinitrophenol, a well-known explosive, in an alleged novel form.

Being an ingredient of explosives, it is apparent that it would be advantageous at times to have the diazodinitrophenol in the form of discrete free flowing particles. As stated by applicant, page 2, second paragraph of the specification, such products have been described in the prior art, but these products "have consisted of opaque agglomerates of large numbers of small needle-like crystals and/or extremely minute particles.''

Applicant has found that by the addition of certain substances to the reaction mixture from which diazodinitrophenol is to be crystallized, the type of crystal growth is controlled so that there are obtained individual crystals having such dimensions as to promote rapid settling from suspension and (which have) the property of free flow in the dry state.'' The specification describes the substances useful to control crystal growth of diazodinitrophenol as certain specified triphenyl-methane dyes (page 4) which must be added in a particular manner in the reaction procedure (page 5, second paragraph). The obtained product is described as comprising:

> ''clear green tabular crystals having on the average, dimensions of about 0.060 mm. by 0.033 mm. by 0.013 mm.''

The accompanying drawings are stated to illustrate the shape of these crystals.

The novelty of applicant's procedure is conceded and claims 7 to 11 which define the same have been allowed thereon. Claims 2 to 6, defining the product as being in the form of tabular crystals containing the specified type of crystallization control ingredient have also been allowed.

The claims under rejection do not include the presence of the control ingredient in the crystals. These claims have been rejected as being incomplete and as not properly defining the invention.

It should be noted that the crystals described by applicant are colored because of ''the color imparted by the absorbed dye (which) permits the identification of the crystals''. Nowhere in the specification is there described a product free from absorbed dye. Nowhere in the description does applicant teach how to obtain the desired form of crystal by means or substance other than the triphenyl-methane dyes. It is submitted, therefore, that the allowed claims are commensurate with applicant's contribution to the art.

Applicant is not the first to have conceived the desirability of having diazodinitrophenol in the form of free-flowing particles. Nor did the art need any teaching that tabular crystals of a chemical product would have this desired property since numerous substances have been prepared in the form of tabular crystals as illustrated, for instance, by Kilbourne. Applicant has taught the art one way of obtaining diazodinitrophenol in the form of tabular crystals: namely, by the addition of triphenylmethane dye, and claims thereto stand allowed.

Applicant points out that the Kilbourne et al. patent contains a claim to:

> "tabular crystals of anhydrous disodium phosphate"

and that applicant's claims at issue are similarly drawn. Although the Office is in no way bound by the previous allowance of a certain type of claims in another patent, the Examiner has pointed out wherein the present disclosure is not on a par with that of Kilbourne to support the same type of claim. Kilbourne taught the art as to disodium phosphate, a tabular crystal could be obtained in several ways, in at least one of which the presence of the control ingredient was not necessary. It is apparent that the two cases are not comparable.

As was stated by the Court in General Electric v. De Forest, 17 F. (2d) 90:

> "Every invention contains two elements: (1) An idea conceived by the inventor; (2) an application of that idea to the production of a practical result. Neither of these elements alone is sufficient."

In the instant case the concept of any particular chemical compound in the form of tabular crystals is not novel. The execution of that concept by finding the means whereby it could be accomplished is applicant's invention and is defined only by the claims allowed.

For the foregoing reasons the rejection of claims 1 and 14 is deemed sound and should be affirmed.

Respectfully,

I. Goldstein,
Acting Examiner, Division 43.

BMK:pef

Paper No. 15.

Appeal No. 44,302

Hearing:
Jan. 28, 1943.

Stamped in Center of page: U. S. Patent Office Board of Appeals Feb 24 1943 Mailed; immediately to right of stamp: NLFD

In the United States Patent Office
Before the Board of Appeals

Ex parte Frederick M. Garfield and Herman W. Dreher.

Application for Patent filed July 3, 1940.

Serial No. 343,706. Manufacture of Diazodinitrophenol. Mr. Joseph Fleischer for applicants.

This is an appeal from the final rejection of claims 1 and 14.

Claim 1 is typical.

> Claim 1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces.

The references relied upon are:

Alexander	2,103,926	Dec. 28, 1937
Hancock ....Re.	19,645	July 16, 1935
Kilbourne	1,962,080	June 5, 1934

The invention relates to a crystalline diazodinitrophenol in which the crystals are tabular in form, characterized by a pair of parallel substantially rectangular faces. In this form the crystals have the characteristics of being free flowing and stable. The problem of forming diazodinitrophenol in a form in which it would be free flowing has claimed the attention of investigators for some time past and appellant accomplished his result by adding certain triphenyl-methane dyes to the reaction mixture whereby crystal growth is controlled so as to produce free flowing diazodinitrophenol in the form of tabular crystals containing an adsorbed triphenyl-methane dye.

Claims to the method and to the product last referred to have been allowed. Appeal was taken from the action of the Examiner's refusal to allow claims to the tabular form, but which do not include the adsorbed dye.

The claims stand rejected as being incomplete and as not properly defining the invention. We understand the rejection to mean that claims 1 and 14 do not properly define the product produced by appellant as disclosed in the specification.

It is to be noted that the claims call for the compound in a certain crystalline form. As pointed out by the Examiner, diazodinitrophenol is not new with this applicant, since such product has before been produced in crystalline form (acicular) and has been produced in several forms (grains, pellets) in which it is free flowing. The advantage of having crystals in tabular form was hardly a patentable concept, since this crystalline form has been recognized as imparting free flowing characteristics, as shown by Kilbourne, in connection with anhydrous disodium phosphate.

The question presented by this appeal appears to be whether appellant invented a composition consisting of diazodinitrol and a triphenyl-methane dye wherein the composition is in tabular crystalline form, as the Examiner seems to contend (which the claims before us obviously do not properly define), or whether appellant also invented diazodinitrophenol in tabular form, and nothing more, insofar as the product is concerned.

Appellant does not deny that his crystals contain adsorbed dye, but urges that the amount of crystallization control agent (dye) retained by the crystals is insufficient to impair the explosive properties of the product. We think this argument is quite beside the point insofar as the scope of claims 1 and 14 is concerned. These claims cover diazodinitrophenol in a certain form and no more. The product disclosed is composed of that compound and the crystallization control agent.

We agree with the Examiner that the appealed claims do not properly define the invention in that they are incomplete. The claims as drawn are broad enough, and apparently were intended, to cover tabular crystals of diazodinitrophenol in pure form which appellant did not produce insofar as the record before us is concerned. While the degree of purity of appellant's product may be sufficient for use in detonators and priming compositions, that does not warrant the allowance of broader claims which do not include the control agent. This agent is necessary to crystal formation and is as much a part of the crystal as is the diazodinitrophenol insofar as its composition is concerned.

The decision of the Examiner is affirmed.

J. W. Clift,
Examiner-in-Chief,
C. H. Shaffer,
Examiner-in-Chief,
E. F. Klinge,
Examiner-in-Chief,
Board of Appeals.

Feb. 24, 1943.

Mr. Joseph Fleischer
c/o Western Cartridge Co.
East Alton, Illinois.

EXHIBIT 3.

Tabular Diazodinitrophenol Made With Dye.

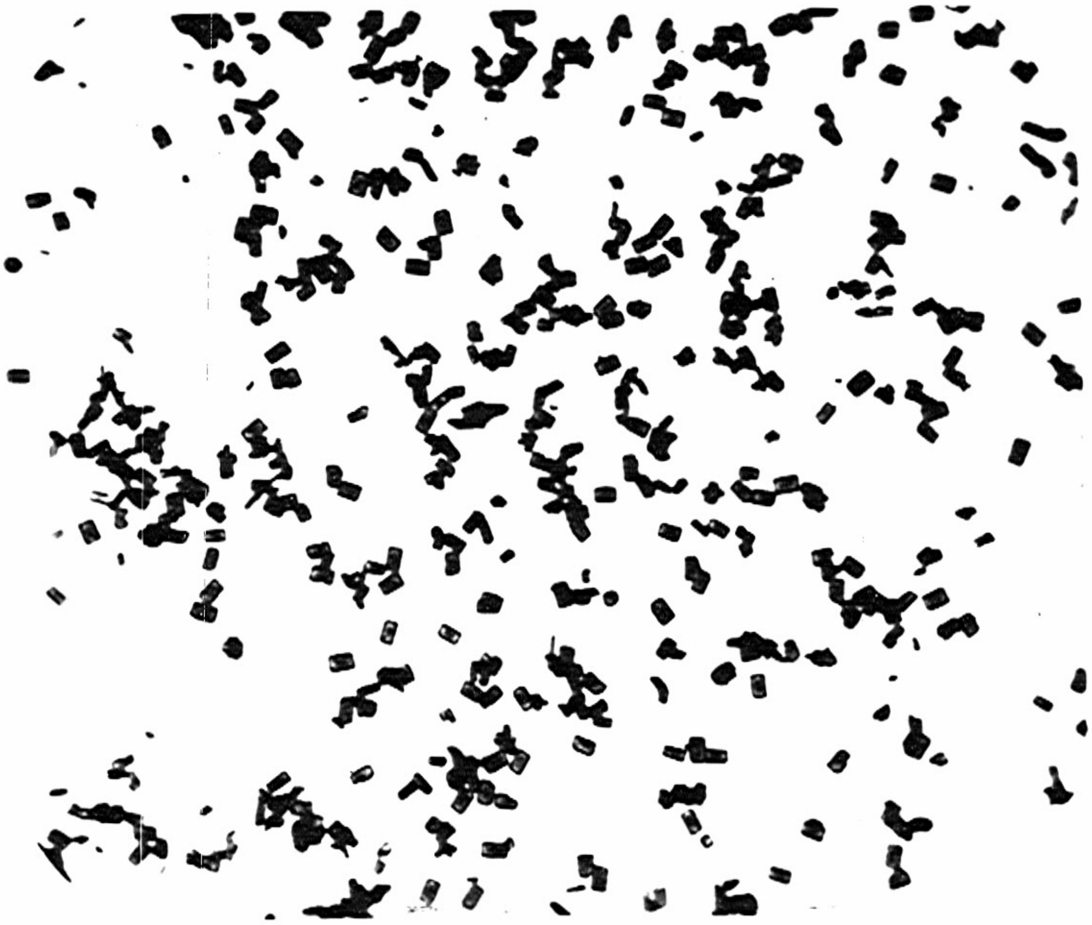


EXHIBIT 9.

Same as Exhibit ~~4~~ 3 After Dusting Test.

EXHIBIT 4.

Nontabular Diazodinitrophenol Made With Same Amount of Same Dye as Ex. 3.

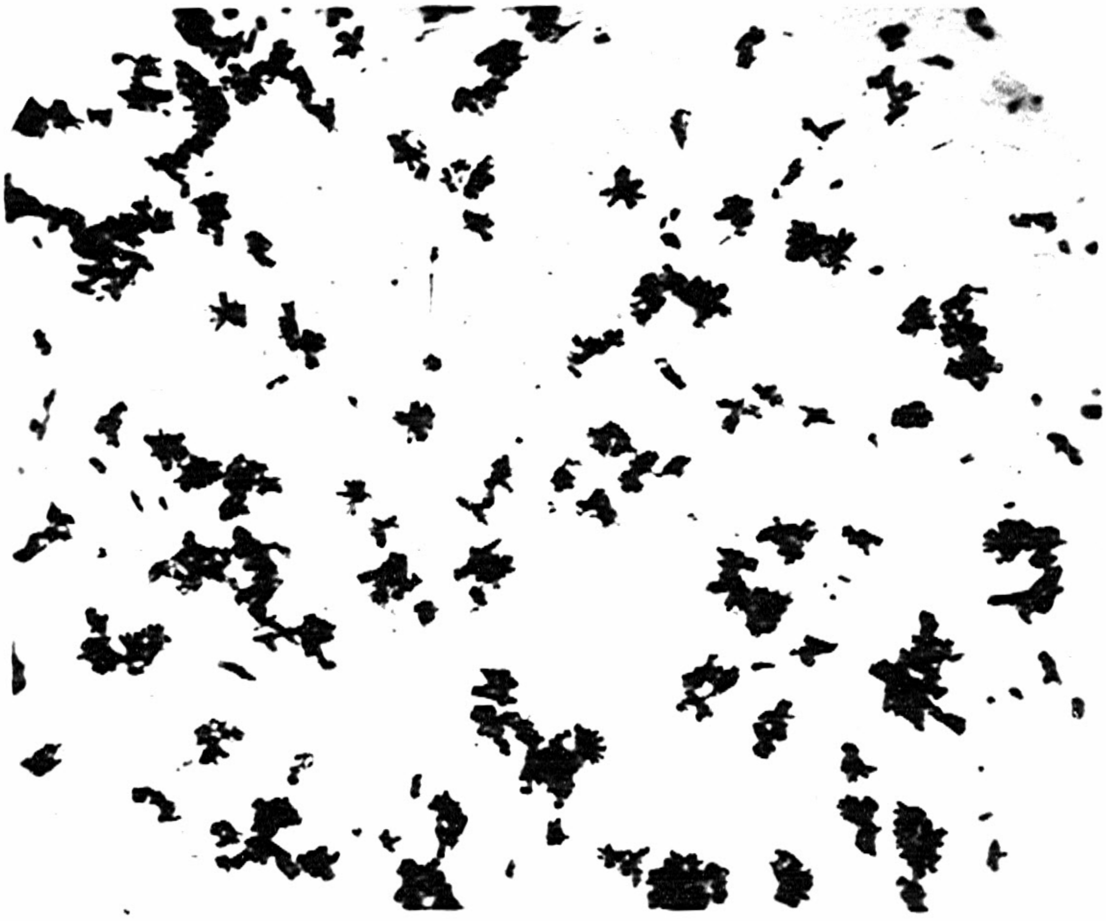


EXHIBIT 8.

Same as Ex. 4 After Dusting Test.

EXHIBIT 5.

Tabular Diazodinitrophenol Made Without Dye.

EXHIBIT 10.

Same as Exhibit 5 After Dusting Test.

EXHIBIT 6.

Needle Diazodinitrophenol.

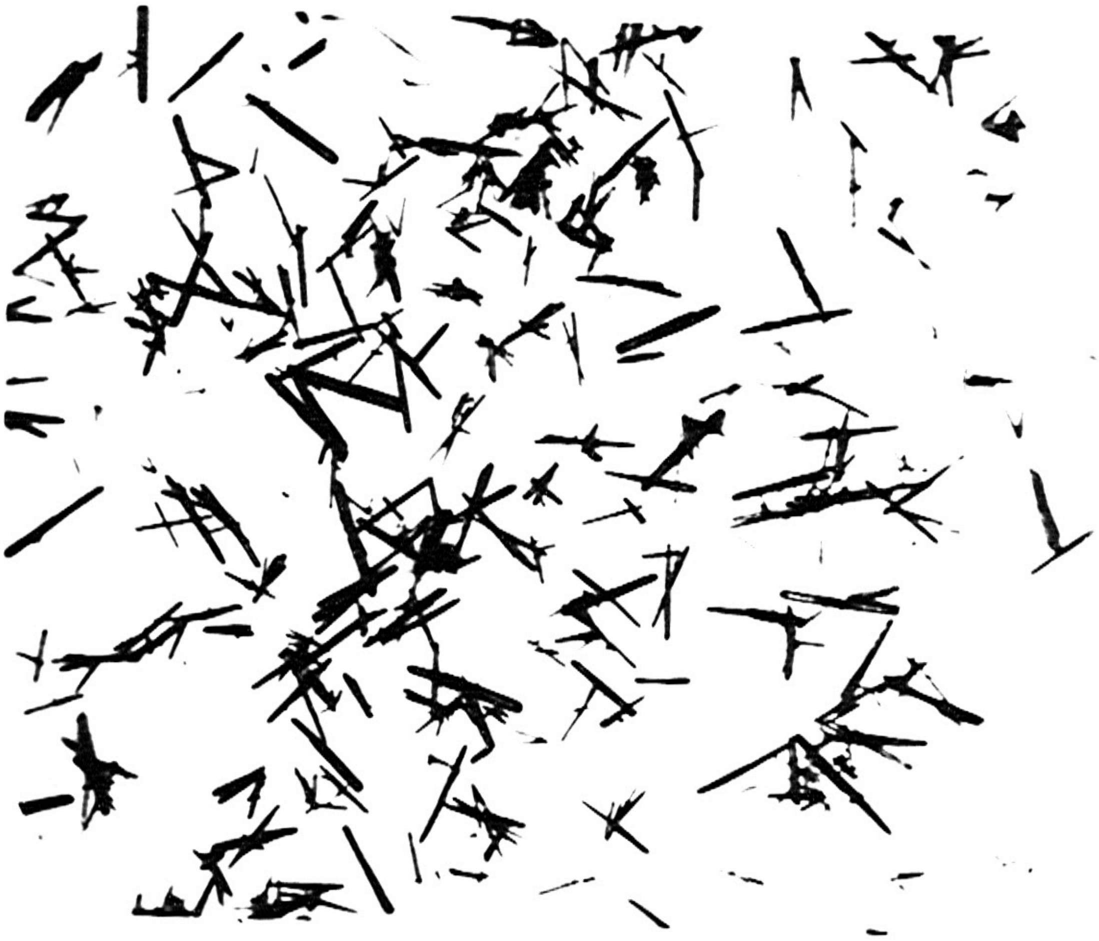
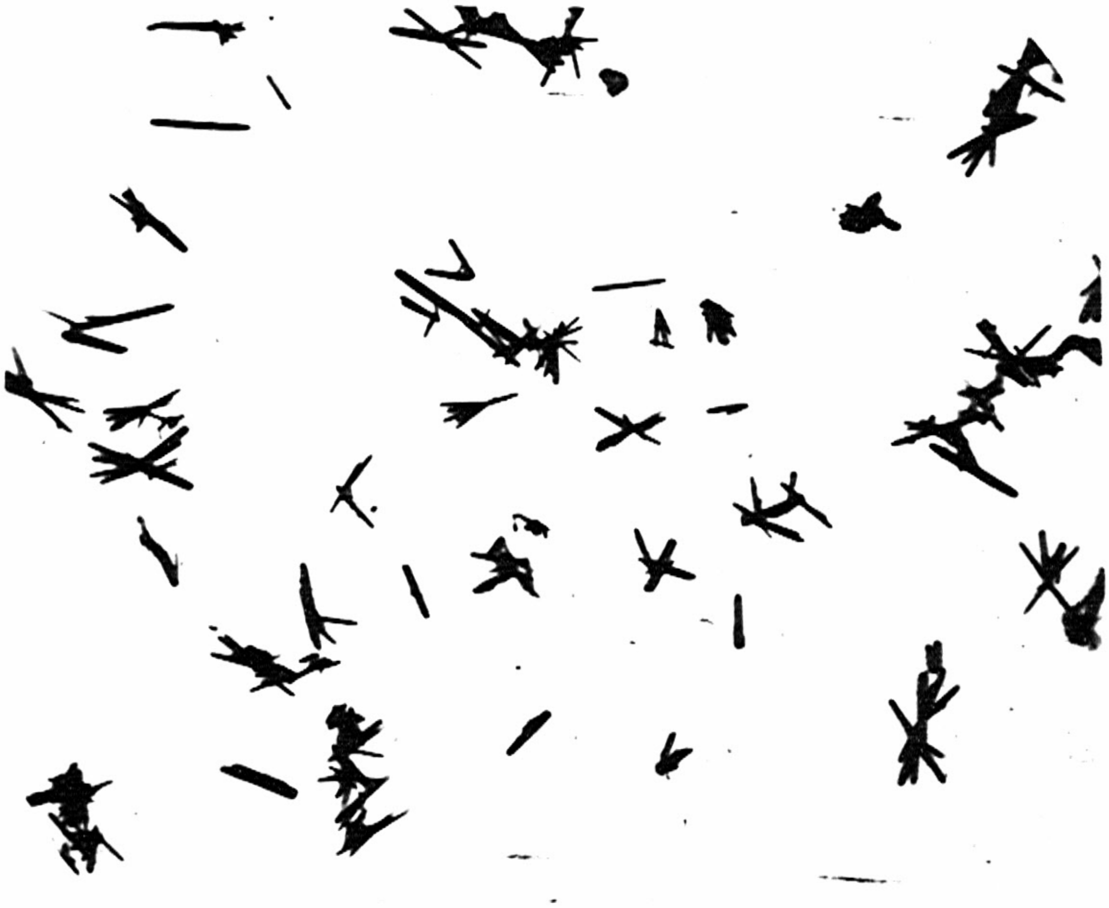


EXHIBIT 7.

Same as Ex. 6 After Dusting Test.



June 5, 1934. K. A. KILBOURNE ET AL 1,962,080

CRYSTALLINE ANHYDROUS DISODIUM PHOSPHATE AND PROCESS OF MAKING SAME

Filed Aug. 29, 1932

Fig. 1.

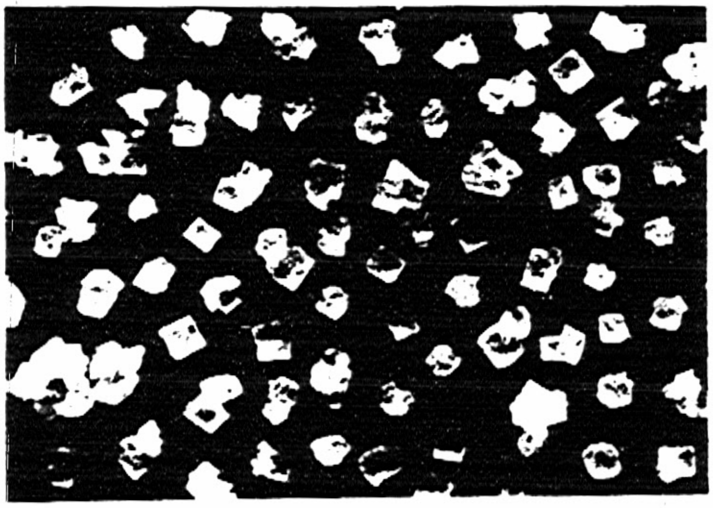
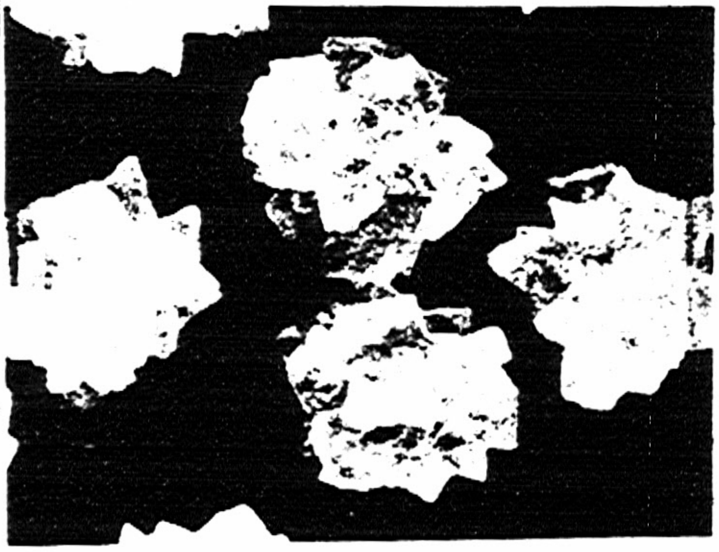


Fig. 2.



Inventors
K. A. Kilbourne
C. F. Booth

By

Herbert J. Chase

Attorney

Patented June 5, 1934 **1,962,080**

UNITED STATES PATENT OFFICE

1,962,080

CRYSTALLINE ANHYDROUS DISODIUM PHOSPHATE AND PROCESS OF MAKING SAME

Kenneth A. Kilbourne and Charles F. Booth, Anniston, Ala., assignors to Swann Research Incorporated, a corporation of Alabama

Application August 29, 1932, Serial No. 630,786

13 Claims. (Cl. 23—107)

This invention relates to an improved form of crystalline anhydrous disodium hydrogen orthophosphate and process of producing same.

Previous methods for the production of anhydrous disodium phosphate consist in the formation of a hydrated crystal, followed by dehydration of such crystals until substantially all water has been removed. Since disodium phosphate usually crystallizes with twelve molecules of water as a hydrate of comparatively low melting point, it is difficult to efficiently dehydrate such crystals because of the very low rate of heat transfer through the crystals or the molten mass and also on account of the large amount of water which it is necessary to evaporate.

In conducting experiments on the production of disodium phosphate, we have discovered that if a pure solution of disodium phosphate is prepared by the neutralization of phosphoric acid with soda ash or caustic soda and the solution thus prepared concentrated by boiling off the water from the solution, crystals of anhydrous disodium phosphate will separate therefrom. We have further discovered that by the addition of tetrasodium pyrophosphate to the neutralized solution, not only is it possible to obtain substantially purer crystals of the anhydrous disodium phosphate, but it is further possible to control the crystal shape. As a result of this discovery, we are enabled to prepare at will crystals of a tabular shape or aggregated crystals, or crystals of an intermediate stage of aggregation.

In order that our invention may be more readily understood, we herewith give the following specific examples of our process and the product therein produced, it being understood, however, that our invention is not to be limited thereby, but is susceptible of various changes and modifications without departing from the spirit thereof.

Example I

Prepare an aqueous solution of disodium phosphate by adding soda ash to phosphoric acid. While various strengths of acid may be used, we prefer to use as concentrated an acid as is economically feasible, since evaporation costs will be lower. A solution prepared in this manner will contain some dissolved carbon dioxide; however, this is easily eliminated during the subsequent evaporation of the contained water. The solution is now evaporated by the application of heat in a crystallizing evaporator of the circulating type, crystallization taking place at temperatures above 96° C. Evaporators suitable for this operation have long been known. For example, evaporators such as are described in U. S. Patents 1,191,108; 905,568; 1,006,823; British Patent 208,503 or in German Patent 381,054 are suitable. The evaporators described in these patents are characterized by a combination of crystallizing and evaporating means in which the flow of the solution is maintained during evaporation in such a manner that evaporation is effected at a place spaced apart from the crystallizing point.

In order that crystallization may be controlled, we find it desirable to add to the solution undergoing evaporation seed crystals of anhydrous disodium phosphate in known manner. Evaporation is conducted by boiling the solution, the boiling point at normal atmospheric pressure being in the neighborhood of 105°–106° C., but may even be lowered to the neighborhood of 96°–97° C. by the application of a partial vacuum to the apparatus in which evaporation is taking place. The crystals obtained in this manner are euhedral tabular crystals, and are shown in Figure 1 on the accompanying drawing, in which the magnification is 10X.

Example II

In order to produce aggregated crystals, we proceed in the same manner as indicated in Example I above, except that we add to the evaporating solution, or to the solution before evaporation, a small percentage of tetrasodium pyrophosphate. The amount added may be in the neighborhood of 0.05% of the pyrophosphate, based upon the weight of disodium orthophosphate in solution. The crystals produced in this example are aggregates of singly terminated crystals, the terminations being domes. These crystals are illustrated by Figure 2 of the drawing, in which the magnification is 8X.

It is possible, by suitably proportioning the amounts of tetrasodium pyrophosphate added to the solution, to control the amount of aggregation of the crystals produced. The proportion of the pyrophosphate employed when it is desired to obtain less aggregation than shown in Figure 2 is less than 0.05%. In general, we believe that if it were possible to measure the amount of aggregation of the crystals caused by the presence of the pyrophosphate, it would be found to be proportional to the amount of pyrophosphate in the solution. However, because of the nature of the cause and effect, we are un-

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able to more specifically describe the nature of this relation.

By increasing the pyrophosphate content of the crystallizing solution above the 0.05% figure mentioned above, it is possible to obtain crystals of anhydrous disodium phosphate containing a small percentage of the pyrophosphate. In this manner it is possible to obtain crystals of the anhydrous salt with ½ to 1%, or even 2 or 3%, contained pyrophosphate. Such a product is valuable in the manufacture of aqueous solutions of disodium phosphate where it is desired to prevent the production of turbid solutions due to dissolved iron and aluminum compounds.

Where amounts of the tetrapyrophosphate of the order of 0.05% are present in the solution, the aggregated crystals formed are probably free of the pyrophosphate compound, at least the amount present is so small as to prevent its recognition by known analytical procedures. Where greater amounts of the pyrophosphate are present in the crystallizing solution, greater amounts come out with the disodium phosphate crystals. Such amounts thus appearing in the crystals are, however, less than the amounts in solution.

The size of the crystals can, of course, be varied in known manner by merely causing the evaporation to continue for a longer or shorter period of time before removing the crystals from the crystallizer.

Optical examination of the crystals shown in Figures 1 and 2 show both to have similar optical properties. They are, biaxial, anisotropic with an optic angle of practically 90° and exhibit the following refractive indices for ordinary light:

X=1.482
Y=1.494
Z=1.512

the error in measurement being of the order of plus or minus 0.003.

The physical properties of both the tabular and the aggregated crystals are such as to render them non-caking and extremely free flowing. Due to the absence of water of hydration in our improved product, a concentrated form of disodium phosphate is available in well-defined crystalline form.

While we have described only a few embodiments of our invention, it will be apparent to those skilled in the art that it is not so limited, but that it is susceptible of various changes and modifications without departing from the spirit thereof, and we desire that only such limitations shall be placed thereupon as may be imposed by the prior art, or as are specifically set forth in the claims.

What we claim is:

1. Process of producing aggregated tabular crystals of anhydrous disodium phosphate which consists in crystallizing the disodium phosphate from a disodium phosphate solution at a temperature of above 96° C., while maintaining a circulation of said solution.

2. Process of producing aggregated crystals of anhydrous disodium phosphate, which consists in crystallizing the disodium phosphate from a solution of disodium phosphate containing a small proportion of tetrasodium pyrophosphate at a temperature of above 96° C., while maintaining a circulation of said solution.

3. Process of producing aggregated crystals of anhydrous disodium phosphate, which consists in crystallizing the disodium phosphate from a solution of disodium phosphate containing at least 0.05% of tetrasodium pyrophosphate based upon the weight of disodium phosphate in said solution, while maintaining a circulation of said solution.

4. As a new product, tabular crystals of anhydrous disodium phosphate.

5. As a new product, aggregated tabular crystals of anhydrous disodium phosphate.

6. As a new product, aggregated tabular crystals of anhydrous disodium phosphate containing a small amount of tetrasodium pyrophosphate added to influence crystal growth.

7. As a new product, aggregated tabular crystals of anhydrous disodium phosphate containing a small amount of tetrasodium pyrophosphate.

8. As a new product, aggregated tabular crystals of anhydrous disodium phosphate containing between ½ and 3 percent of tetrasodium pyrophosphate.

9. As a new product, anisotropic tabular crystals of anhydrous disodium phosphate having the refractive indices:

X=1.482
Y=1.494
Z=1.512

10. As a new product, anisotropic aggregated tabular crystals of anhydrous disodium phosphate having the refractive indices:

X=1.482
Y=1.494
Z=1.512

11. Process of producing aggregated tabular crystals of anhydrous disodium phosphate crystals which comprises crystallizing the disodium phosphate from a disodium phosphate solution at the boiling point at substantially atmospheric pressure and while maintaining a circulation of said solution.

12. Process of producing aggregated tabular crystals of anhydrous disodium phosphate crystals which comprises crystallizing the disodium phosphate from a disodium phosphate solution at a temperature above 96° C. and while maintaining a forced circulation of said solution.

13. Process of producing aggregated tabular crystals of anhydrous disodium phosphate crystals which comprises seeding a solution of disodium phosphate with anhydrous disodium phosphate seed crystals and crystallizing anhydrous disodium phosphate while maintaining a circulation of said solution.

KENNETH A. KILBOURNE.
CHARLES F. BOOTH.

DEFENDANT'S EXHIBIT B.

RECEIVED.
AUG 5 1944

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Dec. 28, 1937. H. B. ALEXANDER **2,103,926**

METHOD OF PRODUCING DIAZODINITROPHENOL AND PRODUCT THEREOF

Filed Aug. 8, 1936

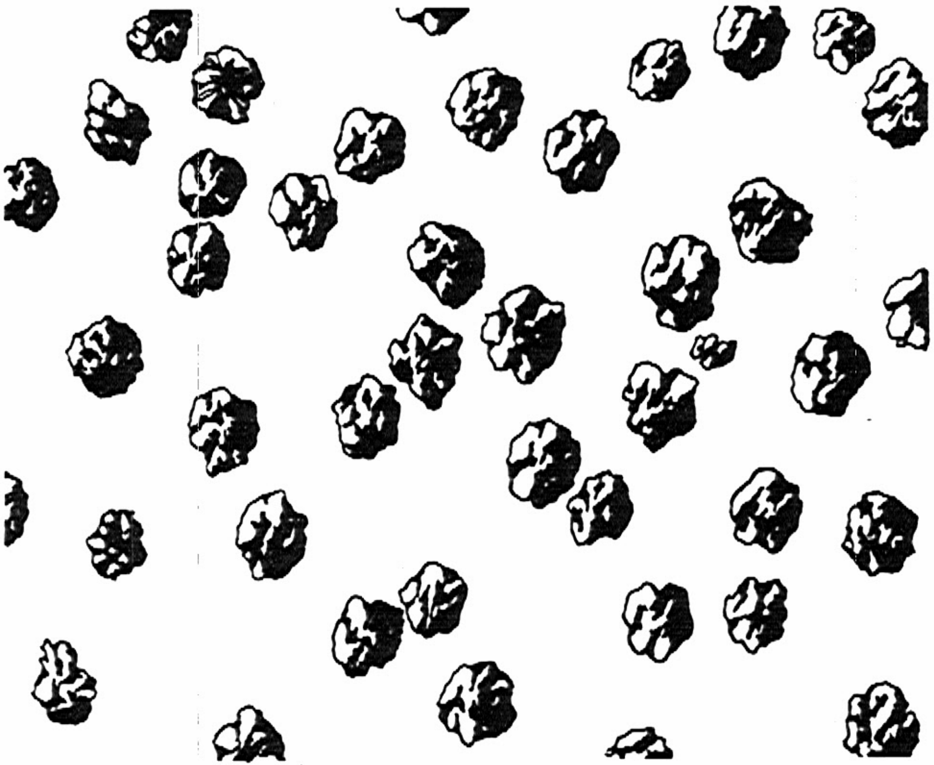


FIGURE 2

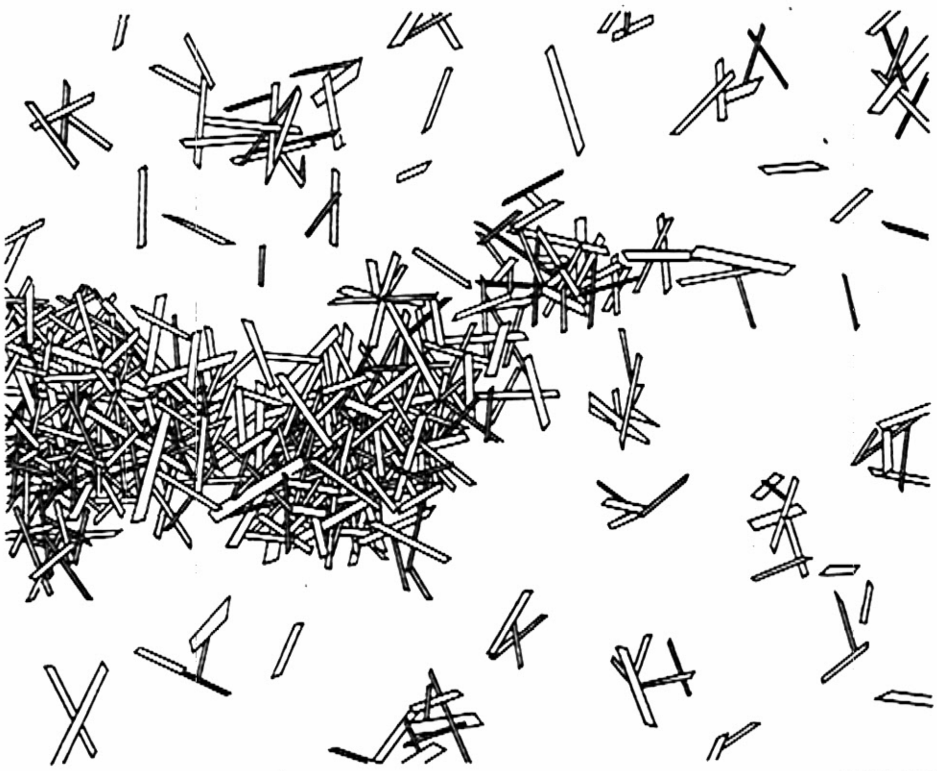


FIGURE 1

INVENTOR

Harvey B. Alexander

BY

Busser + Harding

ATTORNEYS

Patented Dec. 28, 1937 2,103,926

UNITED STATES PATENT OFFICE

2,103,926

METHOD OF PRODUCING DIAZODINITROPHENOL AND PRODUCT THEREOF

Harvey B. Alexander, Kingston, N. Y., assignor to Hercules Powder Company, Wilmington, Del., a corporation of Delaware

Application August 8, 1936, Serial No. 95,014

4 Claims. (Cl. 260—69)

This invention relates to a method for the production of diazodinitrophenol, and more particularly to a method of producing diazodinitrophenol directly in free-flowing form.

Diazodinitrophenol has been known heretofore, and methods for its production have been disclosed. For example, diazodinitrophenol and a method for its production are disclosed in United States patents to Dehn, Nos. 1,404,687, 1,428,011, and 1,460,708. The compound is a very valuable composition for use in blasting caps. However, as generally produced, it is not sandy or free-flowing, and gives great difficulty in loading blasting caps by mechanical means.

Attempts have been made to produce free-flowing diazo grains, e. g., as described in United States Patent No. Re. 19,645 to Hancock and Pritchett, but such has not been wholly satisfactory.

In accordance with my invention a novel method is provided for the production of diazodinitrophenol directly in free-flowing form, adapted to mechanical loading into blasting cap shells.

The method embodying this invention comprises diazotization of a salt of picramic acid by a salt of nitrous acid and an acid, in the presence of a substance adapted to promote the formation of rounded grains of diazodinitrophenol.

Reference is made to the accompanying drawing, in which Figure 1 represents the needle-like crystals of diazodinitrophenol produced by the known processes from relatively pure ingredients, and Figure 2 represents the rounded, free-flowing form of diazodinitrophenol produced by the method in accordance with my invention.

It will be appreciated that the needle-like crystals shown in Figure 1 of the accompanying drawing will mat and pile like a mass of straw, without flowing through holes into a measuring or loading plate, whereas crystals shown in Figure 2 of the accompanying drawing will flow freely through such holes like sand or mercury fulminate. Thus, crystals of the form shown in Figure 1 become impossible to load into blasting caps by the usual procedure.

With the use of a relatively pure salt of picramic acid, e. g., sodium picramate, a relatively pure sodium nitrite, and relatively pure sulphuric or hydrochloric acid, diazodinitrophenol is produced in the crystal form shown in Figure 1 of the accompanying drawing.

I have found, however, that when certain substances are added to the reaction mixture which produces diazodinitrophenol, rounded, sandy, free-flowing grains of diazodinitrophenol are produced in the shape shown in Figure 2 of the accompanying drawing.

I have found that addition to the reaction mixture comprising a salt of picramic acid, a soluble metal nitrite, and a strong acid, of pyrogallol, hydroquinone, phloroglucinol, catechol, resorcinol, p-methylaminophenol, 3,5-dinitrocatechol, p-aminophenol, isopicramic acid, dinitroresorcinol, o-aminophenol, p-sulfanilic acid, o-nitranilin, β-naphthol, nitrosonaphthol, methyl orange, hydroxylamine, 3,5,3′,5′-tetranitro-2, 2′-dihydroxyhydrazobenzene, o-phenylenediamine, p-phenylenediamine, 2,4-diamino-6-nitrophenol, 2,4,6-trinitrobenzoic acid, 2-amino-4-nitrophenol, 2,4-dinitrophenol, trinitro-m-cresol, p-nitranilin, or a substance of unknown composition formed by decomposing diazodinitrophenol by alkali, or mother liquors from the manufacture of sodium picramate, or substances present in the mother liquors from the manufacture of sodium picramate, or a mixture of any of the above, in various amounts, depending upon the particular compound employed, produces diazodinitrophenol in the shape of rounded, free-flowing crystals of the shape shown in Figure 2 of the accompanying drawing.

I have been unable to determine any generic term or group which comprises compounds having the property of producing diazodinitrophenol in free-flowing form, under the conditions of reaction stated, particularly since I have found that certain organic substances which, by analogy, should act in the same manner, do not so act. Examples of the latter are methylene blue, 3,5,3′,5′-tetranitro-2, 2′-dihydroxyazobenzene, 3,5,3′,5′-tetranitro-2,2′-dihydroxyazoxybenzene, picric acid, 3,5-dinitro-benzoic acid, gum arabic, phenol, anilin, and eosin.

As illustrative of the practical application of the method in accordance with my invention, the following examples are given, all of which produce diazodinitrophenol in free-flowing form:

Example I

About 21 parts by weight of recrystallized sodium picramate and about 6.8 parts by weight of relatively pure sodium nitrite were added to about 300 parts by weight of water in a vessel having a cooling jacket, the slurry or suspension stirred and the temperature maintained at about 14.5° C. to about 15.5° C., then about 0.4 part to about 0.04 part by weight of pyrogallol in 10 parts by weight of water and one drop of an aqueous 20% caustic soda solution added, then about 50 parts by weight of 20% sulfuric acid added at the rate of about 20 drops per minute. After all the sulfuric acid is added, the diazodinitrophenol is isolated in a free-flowing form by filtration, and purified by washing with cold water.

Example II

The same method is used as in Example I, but the pyrogallol is replaced by hydroquinone, phloroglucinol, catechol, or resorcinol.

Example III

The same method is used as in Example I, except that the sodium hydroxide is omitted.

Example IV

The same method is used as in Example I, except that 0.4 part by weight of dinitroresorcinol is used in place of the pyrogallol.

Example V

About 4.2 kg. of pure, dry sodium picramate hydrate was placed in a glass-lined tank, fitted with an agitator, and about 40 liters of water added. Then about 1.4 kg. of pure sodium nitrite dissolved in 5 liters of water was run into the tank. To this slurry in the tank was then added 1.5 liters of a solution of decomposed diazodinitrophenol prepared by adding gradually over a period of 3 hours about 160 cc. of 10% sodium hydroxide to 42 g. of pure diazodinitrophenol. The slurry was then diluted with water to about 52.7 liters, and agitation started. The temperature was adjusted to about 14° C. and there maintained during the reaction. About 6.7 kg. of approximately 6 N hydrochloric acid was added at the constant rate of about 23.4 cc. per minute, so that diazotization, as shown by the starch iodide test for nitrous acid, was complete in about 275 minutes. Agitation was then stopped and the charge in the tank transferred to a suction filter, the mother liquor drawn off, and the granular diazodinitrophenol produced was washed with cold water to free it from impurities.

Example VI

The same method is used as in Example V, except that, instead of adding the decomposed diazodinitrophenol solution, 6.7 kg. of mother liquor from the manufacture of sodium picramate (by reduction of sodium picrate by sodium sulfide) was added before starting the diazotization.

Example VII

The same method is used as in Example V, except that, instead of the decomposed diazodinitrophenol, about 4.2 g. of hydroquinone was added to the slurry before starting the addition of the hydrochloric acid.

The procedures given in the above illustrations by way of example, may be varied widely without departing from this invention. Likewise, various picramates, nitrites, and acids may be used in varying proportions, and conditions of diazotization, e. g., temperature, concentrations, etc., may be varied widely within operative limits.

The method in accordance with this invention is not dependent upon the use of any particular form of apparatus, means for cooling, stirring, or the like.

What I claim and desire to protect by Letters Patent is:

1. Method of producing free-flowing diazodinitrophenol which includes reacting a salt of picramic acid, a soluble nitrite, and a strong acid in the presence of mother liquor derived from the manufactue of picramic acid.

2. Method of producing free-flowing diazodinitrophenol which includes reacting a salt of picramic acid, a soluble nitrite, and a strong acid, in the presence of a substance produced by decomposition of diazodinitrophenol by an alkali.

3. Method of producing free-flowing diazodinitrophenol which includes reacting a salt of picramic acid, a soluble nitrite, and a strong acid in the presence of mother liquor obtained from the manufacture of sodium picramate.

4. The method of producing free-flowing diazodinitrophenol which includes reacting a salt of picramic acid, a soluble nitrite and a strong acid in the presence of an added substance capable, under the conditions of diazotization, of controlling the crystallization of the diazodinitrophenol formed so as to produce rounded, free-flowing particles of diazodinitrophenol; said added substance being selected from the group consisting of pyrogallol, hydroquinone, phloroglucinol, catechol, resorcinol, p-methylaminophenol, 3,5-dinitrocatechol, p-aminophenol, isopicramic acid, dinitroresorcinol, o-aminophenol, p-sulfanilic acid, o-nitranilin, B-naphthol, nitrosonaphthol, methyl orange, hydroxylamine, 3,5,3',5'-tetranitro-2,2'-dihydroxyhydrazobenzene, o-phenylenediamine, p-phenylenediamine, 2,4-diamino-6-nitrophenol, 2,4,6-trinitrobenzoic acid, 2-amino-4-nitrophenol, 2,4-dinitrophenol, trinitro-m-cresol, p-nitranilin, a substance of unknown composition formed by decomposing diazodinitrophenol by alkali, and a substance of unknown composition present in the mother liquors from the manufacture of sodium picramate.

HARVEY B. ALEXANDER.

Reissued July 16, 1935 DEFENDANT'S EXHIBIT C. Re. 19,645

UNITED STATES PATENT OFFICE

19,645

METHOD FOR PRODUCING DIAZODINITROPHENOL AND THE PRODUCT THEREOF

Roy S. Hancock, Cambridge, Mass., and Leslie C. Pritchett, Kenvil, N. J., assignors to Hercules Powder Company, Wilmington, Del., a corporation of Delaware

No Drawing. Original No. 1,952,591, dated March 27, 1934, Serial No. 620,529, July 1, 1932. Application for reissue May 22, 1935, Serial No. 22,814

6 Claims. (Cl. 260—69)

This invention relates to a method for the production of diazodinitrophenol and more particularly to a method whereby diazodinitrophenol may be produced directly in a free-flowing form.

Diazodinitrophenol has been known heretofore and methods for its production have been disclosed. Thus, for example, diazodinitrophenol and a method for its production are disclosed in the United States patents to Dehn Nos. 1,404,687, 1,428,011 and 1,460,708. The compound constitutes a very valuable detonating material for use, for example, in charging blasting caps. However, as directly produced by methods heretofore known, it is not free-flowing and hence cannot be readily loaded.

Now in accordance with this invention a novel method is provided for the production of diazodinitrophenol and one in its more specific embodiment of which free-flowing diazodinitrophenol may be prepared directly.

Having now indicated in a general way the nature and purpose of this invention, we will proceed to a more detailed description thereof from the broad and from the more specific standpoints.

From the broad standpoint the method embodying this invention involves the diazotization of a salt of picramic acid by a salt of nitrous acid and an acid. Thus, broadly, this invention involves the diazotization of a picramate by a nitrite and an acid.

More specifically, and especially where the direct production of diazodinitrophenol in a free-flowing form is desired, the acid will be added gradually at a slow rate, or a portion of the acid may be added rapidly at the start with gradual addition of the balance. The rate of addition of the acid, where such is added gradually, following the more specific procedure in accordance with this invention, will depend upon temperature and other variables obtaining during the addition.

In the practical adaptation of this invention any suitable salt of picramic acid, or picramate, may be used. Thus, for example, sodium picramate, ammonium picramate, magnesium picramate, etc., may be used, one being substitutable for the other in equivalent quantities. Likewise, various salts of nitrous acid, or nitrites, may be used, such, for example, as sodium nitrite, potassium nitrite, barium nitrite, etc., being used in substitution for one another in equivalent quantities.

The acid may be any suitable acid for effecting diazotization of the picramate and nitrite, as, for example, hydrochloric acid or equivalent amounts of sulphuric, phosphoric, acetic, etc., acids.

In carrying out the method the picramate and the nitrites will be in solution or in suspension, or partly in solution and partly in suspension in water. Thus, for example, a suspension of sodium picramate with a concentration as high as 9.7% has been successfully diazotized by the method of this invention without indication that the figure is in any way limiting. The solution or suspension will desirably be substantially neutral, and the concentration of the salts, while they may vary widely, will be such desirably as to give efficient and economic results. The temperature during the diazotization will be controlled and will desirably be maintained, by any suitable means, within about the range 10–16° C. Where the acid is added gradually for the production of diazodinitrophenol in a free-flowing form, the addition will be at a relatively slow rate, say, for example, at the rate of about 0.001 gram HCl per minute per gram of sodium picramate, or at equivalent rates for other acids and/or other picramates. As will be understood, however, the most desirable rate of addition of the acid in any given case will depend upon the temperature and other variables, but will readily be determined in a given case by those skilled in the art. Alternative to adding all of the acid gradually a part, say about 25%, may be added rapidly and the balance added gradually.

As illustrative of the practical application of the method in accordance with this invention, for example, about 61.2 pounds (dry weight) of water wet sodium picramate, from which foreign material has been removed, as by screening, say through a 40 mesh screen, is charged into a wooden diazotization tub, fitted with an agitator, with about 90 gallons of water. About 19 pounds of sodium nitrite dissolved in 10 gallons of water are then run into the tub and the agitator started. The temperature is adjusted, by any suitable means, for example, by the addition of ice to say about 15° C. About 40 liters of 1:1 hydrochloric acid are then added, preferably at a rate such as to require about 300 minutes for the addition of the acid. During the course of the run the temperature is maintained at about 15–16° C. by suitable means, as by the addition of ice as required. About five minutes after completion of the addition of the acid the solution is tested with, for example, potassium iodide-starch paper and if a strong positive test is obtained an excess of nitrous acid in the solution is indicated which shows that the diazotization of the sodium picramate has been completed. The agitation is then stopped and the charge dumped, for example, into a suction filter, the mother liquor drawn off and the diazodinitrophenol washed with water to free it from impurities.

Following the above procedure the diazodinitrophenol produced will be in the form of relatively smooth, substantially round or pellet-like discrete particles and will be free flowing without any subsequent treatment and free-flowing diazodinitrophenol may be obtained by varying the above procedure to an extent by adding part of the acid, say for example, 10 liters, quickly at the start with gradual addition of the balance. Where the diazodinitrophenol is not desired in a free-flowing form the acid may be added without any special attention to the rate of addition.

As has been indicated, the procedure given in the above illustration by way of example, may be widely varied without departing from this invention. Likewise, various picramates, nitrites and acids may be used in varying proportions and the conditions of diazotization, as temperature, etc. may be widely varied within operative limits.

As will be obvious, the method in accordance with this invention is not dependent upon the use of any particular form of apparatus, means for cooling, or the like.

What we claim and desire to protect by Letters Patent is:

1. The method for producing diazodinitrophenol in a free flowing form which includes diazotizing a picramate by the gradual addition of an acid thereto in the presence of a nitrite and water.

2. The method for producing diazodinitrophenol in a free flowing form which includes gradually adding an acid to a substantially neutral mixture of a salt of picramic acid and a salt of nitrous acid and water.

3. The method for producing diazodinitrophenol in a free flowing form which includes diazotizing a picramate by the gradual addition of an acid thereto in the presence of a nitrite and water while maintaining the temperature of the reagents within about the range 10°–16° C.

4. The method for producing diazodinitrophenol in a free flowing form which includes gradually adding an acid to a substantially neutral mixture of sodium picramate and sodium nitrite in water.

5. The method for producing diazodinitrophenol in a free flowing form which includes gradually adding an acid to a substantially neutral mixture of a salt of picramic acid and a salt of nitrous acid in water while maintaining the temperature of the reagents within about the range 10°–16° C.

6. Diazodinitrophenol in the form of free-flowing, relatively smooth, pellet-like, discrete particles.

ROY S. HANCOCK.
LESLIE C. PRITCHETT.

BEST COPY AVAILABLE

Jan. 30, 1940. W. H. WELCH 2,188,771

ELECTRODE

Original Filed Jan. 26, 1934

Fig. 1.

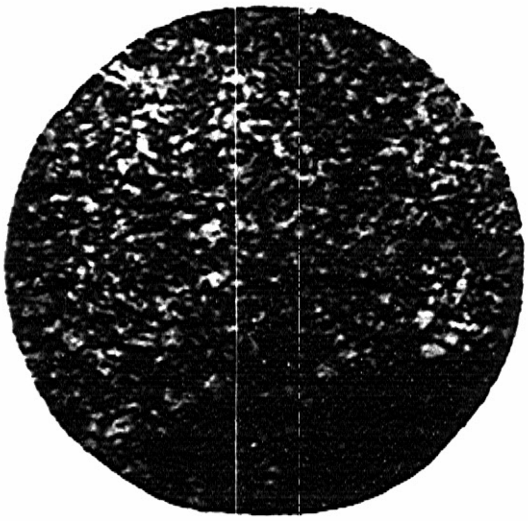


Fig. 2.

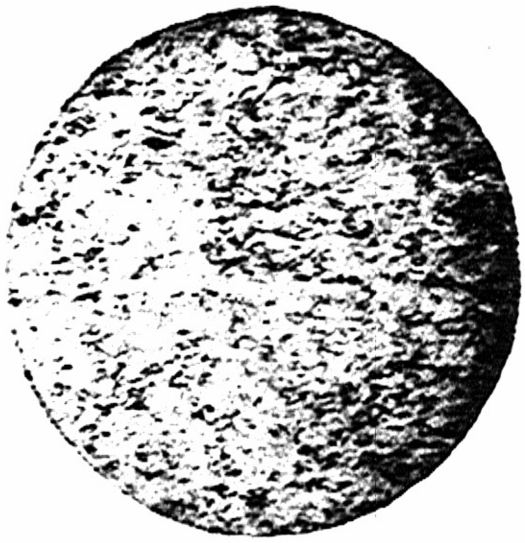


Fig. 3

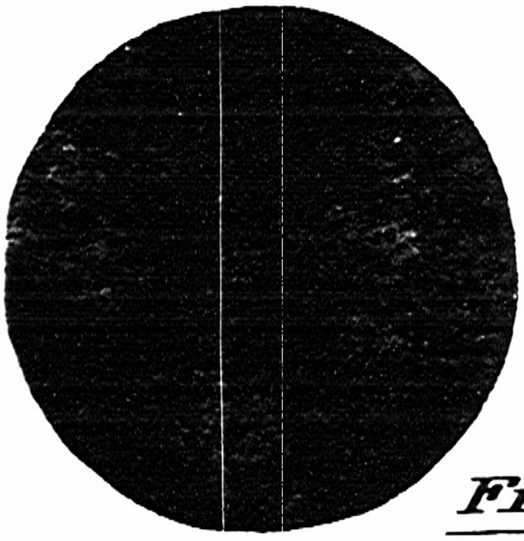


Fig. 4

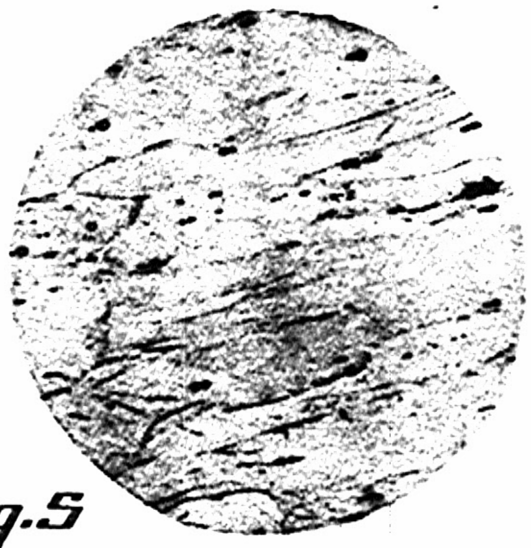
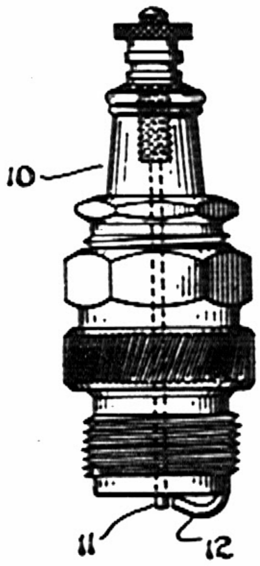


Fig. 5



Inventor

William H. Welch

Albert L. Ely

Attorney

Patented Jan. 30, 1940 **2,188,771**

UNITED STATES PATENT OFFICE

2,188,771

ELECTRODE

William H. Welch, Chicago, Ill., assignor, by mesne assignments, to The Firestone Tire & Rubber Company, Akron, Ohio, a corporation of Ohio

Original application January 26, 1934, Serial No. 708,407. Divided and this application October 7, 1936, Serial No. 104,421

5 Claims. (Cl. 148—32)

This invention relates to electrodes, and more especially it relates to electrode wire that normally is subject to great heat, for example, the electrodes of the spark plugs of internal combustion engines.

Due to severity of conditions incidental to the use of spark plugs, the electrodes of ordinary spark plugs are subject to inter-granular corrosion and rapid disintegration or burning away, which require that periodic adjustment be made to preserve efficient sparking at the gap and which limits the life of the plug in service. Tests show that the ordinary spark plug electrode sponges out during use, and becomes larger in cross section due to inter-granular corrosion, with the result that the granules or crystals on the surface of the electrode lose their cohesion and flake off, thus causing rapid deterioration of the electrode.

The chief objects of the invention are to provide an improved electrode of the character mentioned, that will have relatively long life and that will resist corrosion. Other objects will be manifest.

In achieving the foregoing objects it was found that the structure, particularly grain size and shape of the grains of the electrode wire, was of primary importance, the composition of the alloy being secondary thereto. Best results were obtained where the grain size was relatively small both longitudinally and in cross section, apparently for the reason that since corrosion follows the inter-granular boundaries, the smaller grain size has greater resistance to attack.

This application is a division of my copending application Serial No. 708,407, filed January 26, 1934.

Of the accompanying drawing,

Figure 1 is a photomicrograph of a transverse section of the improved electrode, magnified 200 diameters;

Figure 2 is a view on the same scale as Figure 1, of a longitudinal section of the improved electrode;

Figure 3 is a view on the same scale as Figure 1, of a transverse section of an ordinary prior art electrode;

Figure 4 is a view on the same scale as Figure 1, of a longitudinal section of an ordinary prior art electrode; and

Figure 5 is a side elevation of a spark plug comprising the improved electrode.

It is known that small percentages of certain metals alloyed with nickel retard inter-granular corrosion. Preferably the alloy used in the improved electrode is of the following composition:

		Per cent
Manganese		9.90–4.25
Silicon		.75–1.00
Cobalt	maximum	.25
Carbon	do	.06
Sulphur	do	.03
Iron	do	.25
Nickel		Balance

The percentages may be varied slightly without appreciable detrimental effect This alloy is substantially standard for the usual spark plug electrodes, and no novelty is claimed for it.

In the manufacture of electrode wire from metal of the foregoing composition, the alloy first is cast in ingot form, which ingot is then rolled into a bar and the latter drawn through successively smaller drawing dies until the desired wire size is reached. Between each pass or drawing operation the wire is annealed by heating at a temperature of 1200° F. to 1550° F., the time and temperature of annealing being carefully controlled to prevent grain growth. In order that the minimum grain size may be obtained in the finished product, the reduction in all drawing operations is very carefully controlled. Too great reduction in any of the passes would necessitate excessive annealing, which is objectionable in that it promotes grain growth. Experience has shown that about 25% reduction of the wire on each pass through the dies gives satisfactory results.

While the process may be carried out by the usual method of intermittent drawing and pot annealing, it is particularly adapted for a continuous method of drawing and annealing, the latter step being carried out in an elongated furnace through which the wire is continuously passed.

Just before the final drawing operation, the wire is given a special heat treatment which consists in "soaking" at the critical temperature of recrystallization, within a range of 1200° F. to 1600° F. according to a duration of treatment of 6 hours to 2 hours respectively in the case of treatment of a roll of wire by the pot annealing process, depending upon the amount of wire treated and the container in which it is placed. Preferably the wire is treated about 2 hours at 1550°. In the continuous annealing and drawing process a lesser time of heat treatment is required due to the relatively smaller mass presented and to the relatively larger heater volume. It has been found that if the wire is heated below the proper temperature or for too short a time it would not be properly annealed, and if heated above this temperature or for too long a time a coarse grain would be produced due to combining of smaller grains into larger size particles

The wire is finished in the usual manner and in final form presents a smooth, bright and clean surface free from slivers or flaws.

Referring now to the drawing, Figure 5 shows a spark plug 10 having electrodes 11 and 12, both of which preferably consist of the improved electrode wire. Microanalysis shows the improved electrode wire to be composed of grains of one one-hundredth (1/100) to one three-hundredth (1/300) the grain size of the ordinary electrode wire for the same purpose. The relative smallness of the grains of the improved wire as compared to the grains of prior structures will be apparent from a comparison of Figures 1 and 2 with Figures 3 and 4 respectively. In a grain count made on a photomicrograph of the improved electrode wire at 200 diameters, such as Figure 1, a two-inch diameter circle representing an area of 100 circular mils will contain at least 200 grains. Moreover, the grains of the improved wire are equi-axed, that is, the particle size is substantially the same transversely as longitudinally. It has furthermore been found that electrode wire treated by this improved process has its grain and crystalline structure in equilibrium so that further grain growth and recrystallization within the grain is inhibited when the electrode wire is subjected to elevated temperatures.

The fine grained structure of the wire affords a smooth, bright finish thereon, and makes the wire more resistant to intergranular attack than a corresponding coarse-grained wire. The latter also would have a rough or pebbly surface. Actual tests have shown that the use of the improved electrode greatly prolongs the life of spark plugs in service.

Modification may be resorted to without departing from the spirit of the invention or the scope of the appended claims, which are not limited wholly to the specific construction shown or exact procedure described.

What is claimed is:

1. An electrode consisting mainly of nickel having a grain count of at least 200 in an area of 100 circular mils.

2. An electrode consisting of metal alloy that is mostly nickel, the grain size of said metal being one one-hundredth to one three-hundredth the normal size of the grains of said alloy.

3. An electrode wire consisting essentially of nickel, the grains of which are equi-axed.

4. An electrode consisting mainly of nickel having a grain count of at least 200 in an area of 100 circular mils, said grains being equi-axed.

5. An electrode consisting of metal alloy that is mostly nickel, the grain size of said metal being one one-hundredth to one three-hundredth the normal size of the grains of said alloy, said grains being equi-axed.

WILLIAM H. WELCH.

PLAINTIFFS' EXHIBIT 13.

Patented May 10, 1927. **1,627,863**

UNITED STATES PATENT OFFICE.

ARTHUR S. O'NEIL, OF ALTON, ILLINOIS, ASSIGNOR TO WESTERN CARTRIDGE COMPANY, OF EAST ALTON, ILLINOIS, A CORPORATION OF DELAWARE.

EXPLOSIVE POWDER AND PROCESS OF MAKING SAME.

No Drawing. Application filed December 1, 1924. Serial No. 753,344.

This invention relates to explosives, and more particularly to explosives containing ammonium nitrate as a base.

One of the objects of this invention is to provide an explosive grain which is globular or spherical.

Another object is to provide a globular or spherical explosive grain which is coated to provide a water-proof grain.

Another object is to provide a process for securing definite and uniform granulation of the grains.

Another object is to provide a process for producing uniformly and continuously coated grains.

Another object is to provide a definite progressive explosive mixture.

Further objects will appear from the detail description in which will be illustrated preferred embodiments of this invention; it will, however, be understood that this invention is susceptible of other embodiments. In the following description the invention will be particularly described as applied to the manufacture of explosives containing ammonium nitrate as a base; it will, however, be understood that various features of this invention may be applied to other compounds and mixtures. Ammonium nitrate is employed in a blasting mixture containing a suitable sensitizer, such as nitro-glycerin.

Using ammonium nitrate as an example, a convenient process may be carried out generally as follows: Ammonium nitrate melts at about 158° C. and decomposes at 210° C. In this process the temperature of the salt is raised to a point between 158° C. and 200° C., this temperature being considered practicable to sufficiently liquefy the substance and to be low enough to avoid decomposition. The salt is melted and maintained at the above temperature and the liquid ammonium nitrate poured into a pan or sieve, having predetermined perforations, this pan being elevated over an air shaft. The droplets leaving the pan then fall through the air space, solidify and in falling assume a spherical shape.

At the bottom of the shaft is a tank or other container in which is placed a compound which waterproofs the spheres. This compound may be liquid such as paraffin oil, rock oil, liquid nitro aromatics, such as liquid trinitro-toluene, nitro compounds obtained by nitrating solvent naptha, mononitrobenzoles and toluols, nitro caster oil, nitro bodies obtained by nitrating petroleum fractions, etc., or may be a solid whose melting point is below the melting point of ammonium nitrate, such as dinitro-toluene and trinitro-toluene, nitronapthalenes, nitrobenzoles, petrolatum, paraffine or other waxes. The tank is maintained at a temperature which insures a maximum liquid condition of the coating material and the pellets are received in this tank. When a sufficient quantity is received it is transferred to a heated centrifugal and the excess coating material removed.

Instead of dropping the explosive in the coating medium, this coating medium may be run into the perforated dripping pan with the ammonium nitrate. The explosive body and the coating medium will thus descend together in drops and the coating medium will collect around the explosive drops and form a coating.

An alternative method of waterproofing is to introduce the grains into a revolving barrel or any suitable mixer provided with steam heat and to add to this mixture a compound composed of a polymerizable oil dissolved in a suitable solvent. Chinawood oil is preferred although such oils as Perilla and linseed may be used.

The method of operation is as follows: A suitable quantity of ammonium nitrate in the granular form, preferably as heretofore described, is introduced into the mixing apparatus. Chinawood oil mixed with 50% of carbon tetra-chloride is then poured on the mixture while in motion. Any desired quantity of this solution may be used but it is preferable to use in the neighborhood of one-half to one per cent of the weight of ammonium nitrate. After the pellets are completely coated with the above solution, the heat is turned on and the mixing continued until all of the solvent is evaporated. The temperature is then raised to the neighborhood of 100° C. until the oil is completely polymerized, this requiring from one to two hours at the above temperature and producing a uniformly coated grain, having water resisting qualities.

There is thus formed a globular or spherical explosive grain as distinguished from one with simply rounded corners or edges. This is due to the fact that the grain is a solidified drop which is freely formed while the body is suspended in space and as it falls. The fact that the drop solidified while it is falling in a flowing or fluid condition causes it to become solidified while under surface tension and without restraint; accordingly it assures the natural form of a globule or sphere. Moreover the coating is applied as the explosive is granulated, and since the globules are smooth and not angular a uniform and continuous coating is insured.

In accordance with this invention the granulation is obtained in accordance with the size of the drops, which is determined by the size of the perforations in the pan or sieve. Accordingly a definite and uniform granulation is insured irrespective of any degree of concentration of the explosive compound. The globular grains may, therefore, be made of any desired size or sizes and all globules of the same size will be uniform. The term "drop" as used in the claims is intended to cover a particle derived or formed from a completely molten material.

In accordance with this invention a progressive burning mixture may be obtained and the characteristics of the mixture may be varied by varying the grain sizes. Thus globular grains of varying sizes may be blended, and by varying the proportions of each size in the blended mixture, the desired progressive burning mixture may be obtained. The detonation of ammonium nitrate is the result of an induced wave of detonation from the initiator or sensitizer and the wave is propagated at a diminished rate and intensity as it proceeds. The distance to which this wave travels depends upon the intensity of the induced wave. In other words, the detonation of ammonium nitrate in a body takes place in the immediate vicinity of the initiator and to detonate a quantity of ammonium nitrate it is necessary to mix with it a quantity of a substance which is easily detonated. In this way, the wave is propagated throughout the mass. Therefore, grain size, as such, has no effect on the detonation of ammonium nitrate. The grain size effects the intimacy of the mixture of the sensitizing substance and the ammonium nitrate. The smaller the grains, the more intimate the mixture and vice-versa. The more intimate the mixture, the more rapid the detonation and vice-versa. By controlling the grain size, I then can control the mixture and by controlling the mixture can control the detonation. My method of graining gives pellets of any desired size so that a definite progressive burning mixture may be obtained.

Although this invention has been particularly described with reference to an explosive having ammonium nitrate as a base, it will be understood that certain features thereof may be applied to other explosives and other bases. It will further be obvious that various changes may be made in details without departing from the spirit of this invention; it is, therefore, to be understood that this invention is not to be limited to the specific details described.

Having thus described the invention, what is claimed is:

1. A globular explosive grain freely formed in space from a completely molten material.

2. A solidified explosive drop formed from a completely molten material.

3. An explosive grain which consists of a solidified drop formed from a completely molten composition containing a nitrate.

4. A solidified drop of ammonium nitrate.

5. A coated solidified explosive drop.

6. A coated solidified drop of ammonium nitrate.

7. A solidified explosive drop coated with a water-proofing agent.

8. A solidified explosive drop coated with a hydrocarbon.

9. A solidified explosive drop coated with a nitro-hydrocarbon.

10. The process consisting in causing a completely molten explosive to become solidified in space while under surface tension.

11. The process consisting in causing completely molten explosive drops to become solidified while falling.

12. The process consisting in causing an explosive while in a completely molten state to drop through a sufficient distance to solidify in space.

13. The process consisting in breaking a completely molten explosive into drops and causing the drops to fall and solidify in space.

14. The process consisting in converting an explosive into a completely molten condition, and causing the same to fall as drops and solidify in space.

15. The process consisting in causing completely molten explosive drops to fall and solidify in space.

16. The process of forming globular coated explosive grains consisting in causing the grain body to become solidified while under surface tension and forming a coating thereon.

17. The process of forming globular coated explosive grains consisting in causing explosive drops to become solidified while falling and forming a coating thereon.

18. The process consisting in causing explosive drops to fall into a coating medium.

19. The process consisting in melting an explosive and causing the same to fall as drops and into the coating medium.

20. An explosive consisting of a mixture of solidified explosive drops.

21. An explosive consisting of a mixture of solidified explosive drops and a sensitizer.

22. An explosive consisting of a mixture of solidified explosive drops of progressively increasing sizes.

23. An explosive consisting of globular explosive grains of progressively increasing sizes.

24. An explosive consisting of globular explosive grains comprising ammonium nitrate, said grains being of progressively increasing sizes.

25. The process consisting in forming the explosive into definite drops of progressively increasing sizes.

In testimony whereof I affix my signature this 12th day of November, 1924.

ARTHUR S. O'NEIL.

In the District Court of the United States
for the District of Columbia.

Frederick M. Garfield and Herman W. Dreher and Western Cartridge Company, Plaintiffs, v. Conway P. Coe, Commissioner of Patents, Defendant.	Civil Action. No. 20,979.

FINDINGS OF FACT.

1. This is an action under R. S. 4915 (U. S. C., title 35, sec. 63) in which it was sought to have the Court find that plaintiffs, Frederick M. Garfield, Herman W. Dreher and Western Cartridge Company, are entitled to have issued to them a patent containing claims 1 and 14 of the Garfield and Dreher application No. 343,706.

2. The Garfield and Dreher application discloses the production of tabular crystals of diazodinitrophenol. The process disclosed embodies the use of a dye for controlling the formation of crystals and the application neither discloses the production of such crystals without the use of a dye nor suggests the possibility that they may be so produced.

3. The patent to Kilbourne No. 1,962,080 discloses the production of tabular crystals of anhydrous disodium phosphate and states that such crystals are "extremely free flowing".

4. The patent to Alexander No. 2,103,926 discloses the production of diazodinitrophenol in the form of needle-like crystals and also of pellet-like crystals which are said to be free flowing.

5. The reissue patent to Hancock No. 19,645 discloses the production of diazodinitrophenol in the free flowing form of pellet-like particles.

6. The application here involved does not disclose the production of any tabular crystal which does not contain dye as an ingredient.

7. Claims 1 and 14 of the plaintiffs' application are incomplete and misleading as applied to the process disclosed in such application.

8. Claims 1 and 14 of the plaintiffs' application do not properly define any invention disclosed in such application.

9. The disclosure of the plaintiffs' application does not afford a proper basis for claims 1 and 14 thereof.

CONCLUSIONS OF LAW.

1. The plaintiffs are not entitled to a patent containing either claim 1 or claim 14 of the application here involved.

2. The complaint should be dismissed as to both of the claims involved.

D. W. O'Donoghue,
Justice.

April 5, 1944.

In the District Court of the United States
for the District of Columbia.

Frederick M. Garfield and Herman W. Dreher and Western Cartridge Company,
Plaintiffs,
v.
Conway P. Coe, Commissioner of Patents,
Defendant.

Civil Action.
No. 20,979.

JUDGMENT.

This action came on to be heard at this term and thereupon, upon consideration thereof, it is, this 5 day of April, 1944,

Adjudged that the complaint in this case be, and the same hereby is dismissed with costs against the plaintiffs.

D. W. O'Donoghue,
Justice.

Approved as to Form:
Chas. E. Riordon,
Attorney for Plaintiffs.

In the District Court of the United States
For the District of Columbia.

Frederick M. Garfield, and Herman W. Dreher, and Western Cartridge Company, Plaintiffs, v. Conway P. Coe, Commissioner of Patents, Defendant.	Civil Action. No. 20,979.

NOTICE OF APPEAL.

Plaintiffs, Frederick M. Garfield, and Herman W. Dreher, and Western Cartridge Company, in the above-entitled cause hereby give notice that they appeal from the judgment of the District Court of the United States for the District of Columbia entered therein on the fifth day of April, 1944, such appeal being taken to the United States Court of Appeals for the District of Columbia.

John H. Sutherland,
1004 Market St.,
St. Louis, Mo.

Of Counsel:

Chas. E. Riordon,
852 National Press Building,
Washington, D. C.

In the District Court of the United States
For the District of Columbia.

Frederick M. Garfield, and Herman W. Dreher, and Western Cartridge Company,
Plaintiffs,
v.
Conway P. Coe, Commissioner of Patents,
Defendant.

Civil Action.
No. 20,979.

STATEMENT OF POINTS UPON WHICH PLAINTIFFS INTEND TO RELY ON APPEAL.

1. That Findings of Fact Nos. 6, 7, 8, 9, and that portion of Finding of Fact No. 2 which holds that Plaintiffs' application does not suggest the possibility of producing tabular crystals of diazodinitrophenol without dye, were each and all induced by an erroneous view of the law.

2. That the inclusion of original claim 1 in Plaintiffs' application as filed was:

(a) A suggestion of the possibility of producing tabular crystals of diazodinitrophenol without dye;

(b) A disclosure of a tabular crystal of diazodinitrophenol which does not necessarily contain dye as an ingredient;

(c) A proper basis for claims 1 and 14 of said application; and

(d) A clear indication that the applicants considered the article of manufacture aspect of their invention to derive from and reside in the novel crystal form.

3. That the evidence establishes that the improved results obtained by applicants' product are attributable to

the novel crystal form thereof and not to the presence of any dye.

4. That applicant is entitled to product claims, such as claims 1 and 14, which are not limited by the inclusion of dye.

John H. Sutherland,
1004 Market St.,
St. Louis, Mo.

Of Counsel:
Chas. E. Riordon,
852 National Press Building,
Washington, D. C.

BRIEF FOR APPELLEE

United States Court of Appeals

DISTRICT OF COLUMBIA

APPEAL No. 8815

FREDERICK M. GARFIELD, HERMAN W. DREHER AND
WESTERN CARTRIDGE COMPANY, APPELLANTS

v.

CONWAY P. COE, COMMISSIONER OF PATENTS, APPELLEE

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA

W. W. COCHRAN,
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Attorney for Appellee.

E. L. REYNOLDS,
Of Counsel.

UNITED STA
COURT OF APPEALS FOR
DISTRICT OF COLUMBIA

FILED OCT 6 1944

Joseph W. Stewart

CLERK

INDEX

Page
Introduction 1
Appellants' application 1
Summary of argument 2
Argument 2
Conclusion 7
Authorities cited:
Bijur v. *Rushmore*, 46 App. D. C. 552 6
Hoskins Mfg. Co. v. *General Electric Co.*, 212 F. 422, 427 4
Remington Cash Register Co. v. *National Cash Register Co.*, 67 F (2d) 585 4
Wilson and Forrest v. *Ellis*, 42 App. D. C. 552 6

United States Court of Appeals

DISTRICT OF COLUMBIA

APPEAL NO. 8815

FREDERICK M. GARFIELD, HERMAN W. DREHER AND WESTERN CARTRIDGE COMPANY, APPELLANTS

v.

CONWAY P. COE, COMMISSIONER OF PATENTS, APPELLEE

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA

INTRODUCTION

This is an appeal from the judgment of the District Court of the United States for the District of Columbia (95)[1] dismissing the complaint in an action under R. S. 4915 (U. S. C., title 35, sec. 63), in which the appellants sought to have the Court authorize the issuance of a patent on their application No. 343,706, containing claims 1 and 14 of the said application. The claims in question appear on page 4 of appellants' appendix.

APPELLANTS' APPLICATION

The application here involved (37) relates to the production of tabular crystals of diazodinitrophenol, which substance the appellants' brief, for the sake of brevity, refers to as "dinol" and which will be so referred to herein. The crystals are given the desired form by the inclusion of a crystal growth control agent which is broadly referred to as a dye.

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[1] The numbers in parentheses refer to pages of appellants' appendix.

SUMMARY OF ARGUMENT

1. In order to justify the allowance of a claim to a product an application must contain a disclosure sufficient to teach those skilled in the art how the product can be made.

2. Merely naming a product in an original claim of an application is not a sufficient basis for the allowance of this claim or others similar to it, if the application does not disclose how the product can be made.

3. The appellants' application does not disclose any method of making the product covered by claims 1 and 14.

ARGUMENT

The claims here involved are drawn to dinol "in the form of tabular crystals" or to "tabular crystals of" dinol. Dinol itself, in other crystaline forms was old prior to the appellants' alleged invention, as shown by the patents to Alexander 2,103,926 (80) and Hancock, reissue No. 19,645 (83), and as conceded by appellants' witness Franz (8) questions 16 to 21. The invention claimed here, therefore, is the production of an old substance in crystals of a new form. The question as to the patentability of this idea over the prior art is not involved, the sole issue being whether the appellants' application contains a disclosure which will support the claims.

While it appears to be contended in appellants' brief that a product might properly be claimed even though no method of making it is disclosed, this position is clearly untenable. In order to refute it, it is only necessary to refer to R. S. 4888 (U. S. C., title 35, sec. 33), which provides that "Before any inventor or discoverer shall receive a patent—he shall file in the Patent Office a written description of the same *and of the manner and process of making constructing compounding and using it,* in such full, clear, concise and exact terms as to enable any person skilled in the art or science to which it appertains or with which it is most nearly connected, to make, construct, compound and use the same." [Italics added.]

Even in the absence of this express statutory provision, it is evident that a disclosure of the method of making the product is prerequisite to the grant of a patent on the product. It is the purpose of the patent laws to promote the progress of science and the useful arts, and such progress is not promoted by merely suggesting the possible existence of something, without explaining how it can be produced.

The fact that an original claim recites the product in question is not material. An original claim is a part of the disclosure of the application, but it is effective only for what it actually discloses. In other words, language included in an original claim has exactly the same effect as if it were included in the body of the specification. Merely naming a product in the specification without teaching how to make it, would not support the allowance of a claim to the product; and the fact that the product is named in an original claim instead of in the body of the specification does not alter the situation. If a product could be patented merely by naming it, without explaining how it could be produced, it would be proper to grant patents on such articles as magic carpets and seven league boots. As was said in *Remington Cash Register Co.,* v. *National Cash Register Co.,* 67 F. (2d) 585:

> It is fundamental that the purpose of the disclosure in any patent is to enable the public to practice the invention—i. e., the idea of the invention—after the patent has expired.

Manifestly, this purpose is not fulfilled, where the idea of the invention is a product unless the disclosure would enable the public to make this product.

It remains to be considered whether the appellants' application discloses how to make the product of claims 1 and 14, and it must first be determined what this product is. Claim 1 calls for dinol "in the form of" tabular crystals, and claim 14 for "tabular crystals of" dinol. The only reasonable meaning of this language is that the crystals contain dinol and nothing else, or, at least, nothing else but minor impurities which have no effect on the crystals. In this connection attention is directed to the decision in *Hoskins Mfg. Co.* v. *General Electric Co.,* 212 F. 422, 427, in which it was held that "element of an alloy" and "element formed of an alloy" are identical, that "formed of" is the same as "consisting of" and that consist means "to be composed or made up of" and is a more specific term than "comprising" or "including."

Certainly the language used in the claims is such as, in ordinary usage, would be taken to mean that the crystals contained nothing other than the substance named. A reference to a bar of iron or to iron in the form of a bar would not be understood as including, for example, steel, even though steel frequently contains less than one-half of one percent of carbon. Similarly, such beverages as Coca-Cola, although they consist almost entirely of water, would not be considered as forms of water.

It should also be noted that the application (45) contains allowed claims which differ from those here involved only by reciting that the crystals contain a dye in addition to the dinol. The conclusion is inescapable, therefore, that the appealed claims are directed to crystals which do not contain dye, since, otherwise, there would have been no object in presenting them in addition to the claims which recite the dye.

For the reasons given, it is submitted that appellants' claims 1 and 14 are directed to tabular crystals of dinol which do not contain a dye. The application, however, clearly fails to disclose the production of such crystals. Thus, the trial court, in its second finding of fact (93) found that "the application neither discloses the production of such crystals without the use of a dye nor suggests the possibility that they may be so produced." This finding is amply supported by the record and is in accord with the testimony of appellants' witness Franz, who conceded on cross-examination (30, 31), cross-questions 214, 215, and 218, that the application does not disclose a process of making tabular dinol crystals which do not contain dye.

The appellants' brief appears to argue the case as if the issue were one of undue breadth of the claims. This is not the case. The question is not whether the claims cover too much in addition to what is disclosed, but whether the application discloses anything which will support the claims. If it does not, then, clearly, the claims should not be allowed. It is immaterial how closely the substance disclosed may approach that claimed. In determining the right of a party to make a claim, every limitation of the claim must be regarded as material (*Bijur* v. *Rushmore,* 46 App. D. C. 395; *Wilson and Forest* v. *Ellis,* 42 App. D. C. 552). It is the function of claims to define the applicants' invention rather than to attempt to cover something similar to it which may later be invented. Appellants have not taught the art how to produce tabular crystals of dinol alone and there is thus no consideration for granting them a monopoly on such crystals. What they have actually disclosed is adequately protected by their allowed claims.

CONCLUSION

It is submitted that the lower court was right in holding that the appealed claims are not supported by the disclosure of the appellants' application, and that the decision appealed from should be affirmed.

Respectfully submitted.

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APPELLANTS' REPLY BRIEF.

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA.

NO. 8815.

FREDERICK M. GARFIELD, HERMAN W. DREHER
and WESTERN CARTRIDGE COMPANY,
Appellants,

v.

CONWAY P. COE, Appellee.

Appeal from the District Court of the United States
for the District of Columbia.

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COURT OF APPEALS FOR THE
DISTRICT OF COLUMBIA

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INDEX.

Page

1. Appellants are entitled to patent their product without limitation to the preferred way of constructing it 1

2. This is no attempt to patent an abstract idea..... 2

3. Tabular crystals of diazodinitrophenol are disclosed by the application........................ 2

4. The formed crystals are not affected by dye...... 2

5. Diazodinitrophenol as used in the claims is consistent with its use in the specification............ 5

Conclusion 7

Cases Cited.

Carnegie Steel Co. v. Cambria Iron Co., 185 U. S. 403, 410 5
Century Electric Co. v. Westinghouse E. & M. Co., 191 F. 350, 366 (C. C. A. 8)........................ 6
Criner v. Micro-Westco, 139 F. (2) 681, 687-8......... 5
Dennis v. Pitner, 106 F. (2) 142, 148 (C. C. A. 7)..... 6
Esnault-Pelterie v. U. S., 81 Ct. Cls. 785.............. 6
Hydraulic Press Corp. v. Coe, 134 Fed. (2) 49........ 5
International Cork Co. v. New Process Cork Co., 6 F. (2) 420, 422 (C. C. A. 2).......................... 6
Paper Bag Case, 210 U. S. 405, 418.................. 1

Statutes Cited.

Federal Food, Drug and Cosmetic Act, Title 21, Sec. 321 (g) (j), U. S. Code.......................... 4
R. S. 4888... 1

Miscellaneous.

Department of Agriculture, Food and Drug Administration Bulletin, S. R. A. F. D. No. 2, Rev. 5, Nov., 1936 4
Webster's New International Dictionary, definition of "adsorb" and "iron"......................... 2, 5

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APPELLANTS' REPLY BRIEF.

---

1. Contrary to Appellee's conjecture (Br. p. 3), we do not contend that any one is entitled to patent a new product without disclosing how to make it. We do contend that, having invented a new physical product* and having disclosed the best known way of "constructing, compounding and using it" (R. S. 4888), Appellants are entitled to patent that product without limitation to the preferred way of constructing it. As the Supreme Court said in the Paper Bag Case, 210 U. S. 405, 418:

> ". . . An inventor must describe what he conceives to be the best mode, but he is not confined to that."

---

*Whose "patentability over the prior art is not involved" (Appellee's Br. p. 2).

2. Neither does this case involve an attempt to patent an abstract idea like "Magic Carpets," "Seven League Boots," or "Flying to the Moon." Quite to the contrary, the invention here is a concrete thing whose novel physical and structural characteristics can be delineated with the acme of particularity, distinctness and definiteness in three words—tabular dinol crystals.

3. The claims here involved are addressed to a tangible, concrete, physical thing—tabular dinol crystals—irrespective of how made. They are:

> "1. As a new product, free-flowing diazodinitrophenol in the form of tabular crystals characterized by a pair of parallel substantially rectangular principal faces.
>
> "14. As a new product, tabular crystals of diazodinitrophenol."

That the subject matter thereof is disclosed by the application is not only shown by the testimony (App. 24), but the District Court affirmatively found the fact so to be:

> "The Garfield and Dreher application discloses the production of tabular crystals of diazodinitrophenol" (App. 93).

4. The notion that dye is an "inclusion" (Br. p. 2) in the tabular crystal is erroneous. Dye is definitely and purposely included in the reaction mixture (used in the best known way of constructing the crystals), but whatever dye attaches itself to the crystal is incidental to the process of constructing the crystal. The operation of the dye is described in the patent application as follows:

> ". . . a small amount of an ingredient having the property of being ****ad**sorbed on the crystals as they

---

**"Adsorb" is defined in Webster's New International Dictionary as follows:

adsorb—"To condense and hold by adsorption."

adsorption—"A phenomenon consisting in the adhesion in an extremely thin layer of the molecules of gases, of dissolved substances, or liquids to the **surfaces** of solid bodies, with which they are in contact; distinguished from *absorption*."

> form in the reaction mixture and thereby of exerting a desirable control on the crystal growth. . . . The action of the crystallization controlling ingredient is such as to prevent growth of the crystals into the elongated columnar or acicular type heretofore known and to promote the formation of crystals having dimensions of length and width of the same order of magnitude" (App. 38-39).

Various dyes are "useful as crystal growth control ingredients" (App. 39-40).

Although "the color imparted by the adsorbed dye permits the identification of the crystals" (App. 43), in no substantial sense can the dye be considered an ingredient of the formed crystals. Once the crystals are formed, the presence or absence of a modicum of dye has no practical effect.

> "Q. 134. From that demonstration could you say whether it is the presence of the dye or the shape of the crystals which determines the improvement in the properties? A. I consider it is adequate proof that it is not the dye, the presence of the dye, which confers the desirable properties" (App. 21).
>
> "Q. 210. Do those advantages over, first, the needle type and, second, the ball type obtain whether or not the tabular crystal dinol is made with or without a dye? A. The tabular crystals, both with and without dye, show those properties" (App. 30).
>
> "Q. 216. Do you consider that the (tabular) crystals which contain dye are better in any respect than those (tabular crystals) which do not include it? A. No" (App. 31).

Even if the modicum of dye which attaches itself to the crystals of dinol be considered an impurity, it does not detrimentally affect the dinol, else the product could not have gone into the use it has (App. 15), particularly since tabular dinol is more costly to manufacture (App. 29) than the prior known forms.

After all, the quantity of dye which attaches to the crystals is "very small" (App. 31). As pointed out in our main Brief (p. 27), the amount of dye put into the reaction mixture constitutes less than one-tenth of one per cent thereof. Even if all the dye in the reaction mixture were adsorbed onto the crystals (which it is not), it would amount to less than one per cent (6 grams of 59% dye to 400 grams of dinol—App. 41).

While we know of no "official compendium" or other published standard of purity for diazodinitrophenol, an impurity content on the order of 1% is abundantly within the tolerance ranges recognized by Government Agencies for drugs and foods. For example, common salt may contain up to 2% of impurities (Department of Agriculture, Food and Drug Administration Bulletin S. R. A. F. D. No. 2, Rev. 5, November, 1936). According to the United States Pharmacopoeia,* phenol may contain up to 2% impurities; alcohol 7.7%; glycerine 5%; sodium nitrite 3%**; potassium nitrate, potassium chlorate, cyclopropane 1%; resorcinol 0.5%; water 0.1%. These are but a few that happen also to be used in the explosives industry. We could go on for pages.

When such a degree of impurity is officially tolerated in chemicals intended for human consumption, it is absurd to apply a more rigorous standard to explosives. Woe be to the "soap" that is only 99-44/100% pure!

Even the "bar of iron" or "iron in the form of a bar" selected by Appellee (Br. p. 5) as a yardstick of exactitude, contradicts Appellee's contentions. No one familiar with iron would consider such expressions to imply that the bar was composed of Simon-pure iron. It is common knowledge that iron in every commercial or practical form contains several per cent of carbon.

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*We invoke judicial notice of the United States Pharmacopoeia, which is recognized as an "official compendium" in, and by reference made a part of, the Federal Food, Drug and Cosmetic Act [Title 21, Sec. 321 (g) (j), U. S. Code].

**The tolerated 3% impurity in the sodium nitrite used in Appellants' process amounts to more than five times the quantity of dye put into the batch.

> ". . . Steel and cast iron contain varying amounts of carbon and other substances (esp. silicon, sulphur, phosphorus, and manganese), which greatly affect the properties. The constituents have been named from a physical-chemical standpoint, as: *ferrite,* or pure iron; *austenite* and *martensite,* a solid solution; *pearlite,* the eutectoid alloy of iron and carbon; *cementite,* the carbide FeC; etc. (see these terms)." (Definition of "iron," Webster's New International Dictionary.)
>
> "Steel is a product, or, perhaps, more accurately, a species of iron, refined of some of its grosser elements, intermediate in the amount of its carbon between wrought and cast iron" (Carnegie Steel Co. v. Cambria Iron Co., 185 U. S. 403, 410).

5. In Hydraulic Press Corp. v. Coe, 134 Fed. (2) 49, this Court, in determining whether to accept a highly technical interpretation of words in the claim or a more common one, held:

> ". . . We think therefore that the term must be taken, both from the general patterns and purpose of the claims and from its more ordinary meaning, to have been used in its common, rather than its more technical sense" (l. c. 56).

As pointed out by the Court of Appeals for the Eighth Circuit in Criner v. Micro-Westco, 139 F. (2) 681, 687-8, the words of a patent claim "are used in a practical rather than a purely scientific sense." On the evidence in this record no one can deny that, in the practical sense, Appellants' product is diazodinitrophenol. It is repeatedly so denominated in the specification:

> "The resulting diazodinitrophenol precipitate consists of individual crystals . . ." (App. 38).
>
> "The process . . . permits . . . of precipitated diazodinitrophenol in the form of individual crystals . . ." (App. 39).
>
> "The present procedure results in desirable free-flowing crystals of diazodinitrophenol" (App. 40).
>
> "The diazodinitrophenol crystals were obtained . . ." (App. 41).
>
> "The crystalline diazodinitrophenol . . ." (App. 42).
>
> "The diazodinitrophenol . . . consists of clear green tabular crystals . . ." (App. 42).
>
> "The novel crystalline diazodinitrophenol" (App. 43).

Under the rule that the patent specification is the dictionary by which the meaning of the claims is determined [Dennis v. Pitner, 106 F. (2) 142, 148 (C. C. A. 7); International Cork Co. v. New Process Cork Co., 6 F. (2) 420, 422 (C. C. A. 2); Century Electric Co. v. Westinghouse E. & M. Co., 191 F. 350, 366 (C. C. A. 8); Esnault-Pelterie v. U. S., 81 Ct. Cls. 785], Appellants are clearly entitled to claim their product with the same nomenclature used to describe it in the specification. While we do not contend that the crystals (resulting from Appellants' preferred process) are chemically pure diazodinitrophenol—although they are as pure as anything that has ever been offered in trade under that appellation—the name is technically and scientifically, as well as practically and commercially, correct.

CONCLUSION.

The Commissioner's position that we cannot call our diazodinitrophenol by its right name because it is stained with dye is as absurd as to say we cannot call our suit "wool" because it is dyed; nor our shoes "leather." Then, after the stain to develop the grain, no more the floor could be "wood."

How the damsel fair, who hennaed her hair,
  And proudly assumed a glamorous air
In this game for a name, was appalled
  At the Commissioner wishin' 'er to declare
Being bare of pure hair—she was bald.

Respectfully submitted,

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